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AKRON, OHIO 44315

MECHANICAL RESPONSE AT CRYOGENIC TEMPERATURES OF SELECTED REINFORCED PLASTIC COMPOSITE SYSTEMS

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Paper No. 16

HERBERT F. HARDRATH

Mechanical Response at Cryogenic Temperatures of Selected Reinforced Plastic Composite Systems

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This is an advance copy of a paper accepted by the Society on the basis of an approved summary for presentation at the Seventieth Annual Meeting of the **American Society for Testing and Materials** (1916 Race Street, Philadelphia, Pa. 19103) to be held in Boston, Mass. June 25 — June 30. The paper has not received the Society review necessary for approval for publication, and responsibility for the contents of this advance copy rest solely with the author. Its purpose is to stimulate discussion. Discussion is invited and may be transmitted to the Executive Secretary. The paper is subject to modification and is not to be published as a whole or in part pending its release by the Society through the Executive Secretary.

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1-a

Abstract

A three-year effort associated with the evaluation of structural reinforced plastic materials for application at cryogenic temperatures has recently been completed under the sponsorship of NASA. This work has been performed at Goodyear Aerospace Corporation and directed from the George C. Marshall Space Flight Center.

The primary objective throughout the program has been the assessment of test techniques and test specimens for the development of usable design information covering a variety of composite systems. Test temperatures have been within the range of normal ambient through 4.2 Kelvin of liquid helium. Evaluations were conducted under controlled material characterizations, optimum specimen designs, controlled processing, precise test procedures and the statistical analysis of data.

Essentially, the flat test specimens conformed to the outlines specified in Federal Test Method Standard No.406 and pertinent ASTM Standards. Modifications to these were slight and were made only as required for adjustment to the peculiarities and severities of cryogenic temperatures. A portion of the program was devoted to the validation of flat specimen data within simple structural models.

Mechanical test data encompassed tensile, compression, shear, flexural and bearing properties. The control composite system consisted of the WS1028A epoxy matrix reinforced with S/HTS-20 end glass roving. This roving was arrayed in both unidirectional and bidirectional orientation within the cured laminate.

The matrix of the control composite system is an anhydride cured epoxy accelerated with a tertiary amine (Epon 828/1031, NMA, BDMA). This paper develops a comparison of the mechanical responses of this system to a number of other promising matrices for cryogenic structural applications. In all cases, the reinforcement was held constant at S/HTS-20 end roving. Based upon a literature survey and industry canvas, the following matrix systems were selected:

1. A chemically pure epoxy resin with an aromatic amine curing agent (DER 332/DEH 50).
2. A chemically pure epoxy resin system with a Lewis Acid curing agent (DER 332/BF_3-MEA).
3. A polyester resin system (Selectron 5158/t-butyl perbenzoate)

The first and last of these matrices were utilized as wet systems while the remainder was in pre-preg form.

An evaluation of the various systems tested indicates that the anhydride epoxy matrix develops the most optimum overall properties. The Lewis acid cured epoxy developed comparable properties with the exception of low interlaminar shear strengths. All three alternate systems displayed significant improvements in compressive strengths. Generally speaking, the four systems may be considered for structural cryogenic applications. The choice would be dictated by specific improvements needed in a structure and by economic considerations.

Foreword

This GER[a] was prepared for presentation at the Seventieth Annual Meeting of the American Society for Testing and Materials to be held in Boston, Mass. 25-30 June 1967.

[a]Goodyear Engineering Report

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Introduction

This paper highlights certain achievements of a three-year effort to evaluate structural reinforced plastic materials for application at cryogenic temperatures. The work was sponsored by the National Aeronautics and Space Administration's George C. Marshall Space Flight Center.[a]

The primary objective was to establish test specimens and procedures suitable for developing physical and thermal properties data down to 4.2 Kelvin (liquid helium). The guiding consideration was that data obtained from the tests be of design utility. The evaluations were conducted under controlled material characterizations, optimum specimen designs, controlled and reproducible processing, precise test procedures, and the statistical analysis of data. The composite system specimens were cut from flat laminates, tubes, and rods.

The program was conducted in three one-year phases, the first of which was the development of techniques for cryogenic testing, using essentially flat-type specimens conforming to the outlines specified in Federal Test Method Standard No. 406 and ASTM Standards. Modifications to these were made as required, adjusting for peculiarities and severities of cryogenic temperatures.

The second phase emphasized the validation, in structural models, of the test values derived from the previous simple flat-type specimens. In addition, an assessment of environmental effects upon cryogenic properties was conducted.

The third phase, which is the principal interest of this paper, applied the previously developed test specimens and techniques to a variety of matrix-reinforcement systems. Using an epoxy anhydride, S-glass combination

[a]Final Report NASA-CR-80061, Microfiche NA67-12051: Program for the Evaluation of Structural Reinforced Plastic Materials at Cryogenic Temperatures. Contract NAS8-11070, NASA Scientific and Technical Information Facility, P.O. Box 33, College Park, Md., 207404.

as the control system, a family of resin matrix and reinforcement combinations were tested for cryogenic response.

The program produced results that will contribute significantly toward the advancement of reinforced composites as engineering materials for use at cryogenic temperatures. These results can be summarized as follows:

1. Test specimens of reinforced plastic laminates, usable through the full test range of 298 to 4 K were designed
2. Test techniques were perfected for the assessment of the mechanical and thermal properties throughout the test range
3. Correct modes of failure were achieved.
4. Specimen sizes and test procedures were developed which are practical economically for cryogenic testing
5. Test data scatter was held to a reasonably narrow range
6. The test data have direct design utility
7. Test data developed in the program establish the required engineering base for direct application of reinforced plastic laminates in structural applications at cryogenic temperatures

Test Specimen Design

General

The function of a test specimen is to yield reliable property data that can be used to develop predictions of performance in structures. Specimen design selection is also related to economics so that test costs can be held within practical limits. As a goal, it is desirable that for each property measured a single specimen design be selected that will be applicable to a broad range of materials and test temperatures. The testing of reinforced plastic composites through the cryogenic environments poses real challenges in meeting these guidelines.

Mechanical Properties

General — The standard test specimens and test methods used to determine mechanical properties of plastic materials are contained in Federal Test Method Standard No. 406 and ASTM standards. However, unique circumstances involved in the testing at cryogenic temperatures and the particular problems associated with the testing of the various materials and orientations of this program made the direct use of these standards impractical. The high cost of cryogenic fluids required the use of small cryostats to minimize the fluid consumption. This in turn limited the space for test specimens and test grips and made the gripping system relatively inaccessible. Also, to prevent the excessive heat loss through the test grips and fixtures, these items were kept as compact as possible. However, since the tests were being conducted at cryogenic temperatures, both the loads required to produce failure and the problem of grip slippage increased, putting a greater burden on the test grips and fixtures. This combination of factors dictated the use of small test specimens.

Also, the use of unidirectional glass roving as one of the candidate materials presented a unique testing problem because of its high tensile and compressive strength, and yet low shear and bearing strength. This required special care to see that the tensile and compressive specimens failed as desired and not in shear or bearing.

With these limitations in mind and as a result of information gathered from the literature survey, preliminary testing was conducted which determined the test specimen configurations shown in Figure 1. A brief discussion of the various specimens is presented in the following paragraphs.

Tension Specimen — The problem area for the tension specimen revolved around the unidirectional filament-wound specimen because of its high tensile strength and low shear strength. After much preliminary testing it was found that the specimen shown in Figure 1, with cloth laminate loading pads bonded to the specimen using an Adiprene L-100 and Moca adhesive system with a 160 deg F maximum temperature cure, produced an ultimate failure at all test temperatures.

Compression Specimen — Preliminary compression tests were required to clarify two areas of concern. The first was the verification of the use of compact compression specimens and test fixtures necessary to eliminate the large heat sinks of the standard compression fixtures that require excessive amounts of liquid helium. The second was to determine if the ultimate compressive strength of the unidirectional filament-wound material could be achieved in the tests. Preliminary tests confirmed that both of these were accomplished through the use of the specimen configuration shown in Figure 1.

Interlaminar Shear Strength — The investigation of test methods for the determination of interlaminar shear strength resulted in the use of two methods. The guillotine shear specimen produced test results more applicable to design. However, the flexural shear specimen is more economical.

Flexural and Bearing Strength — The test specimen configurations selected for the flexural and bearing tests were similar to the specimens recommended in Federal Test Method Standard No. 406. The specimens of Method 1031 for the flexural test and Method 1051 for the bearing test proved satisfactory in obtaining the desired results.

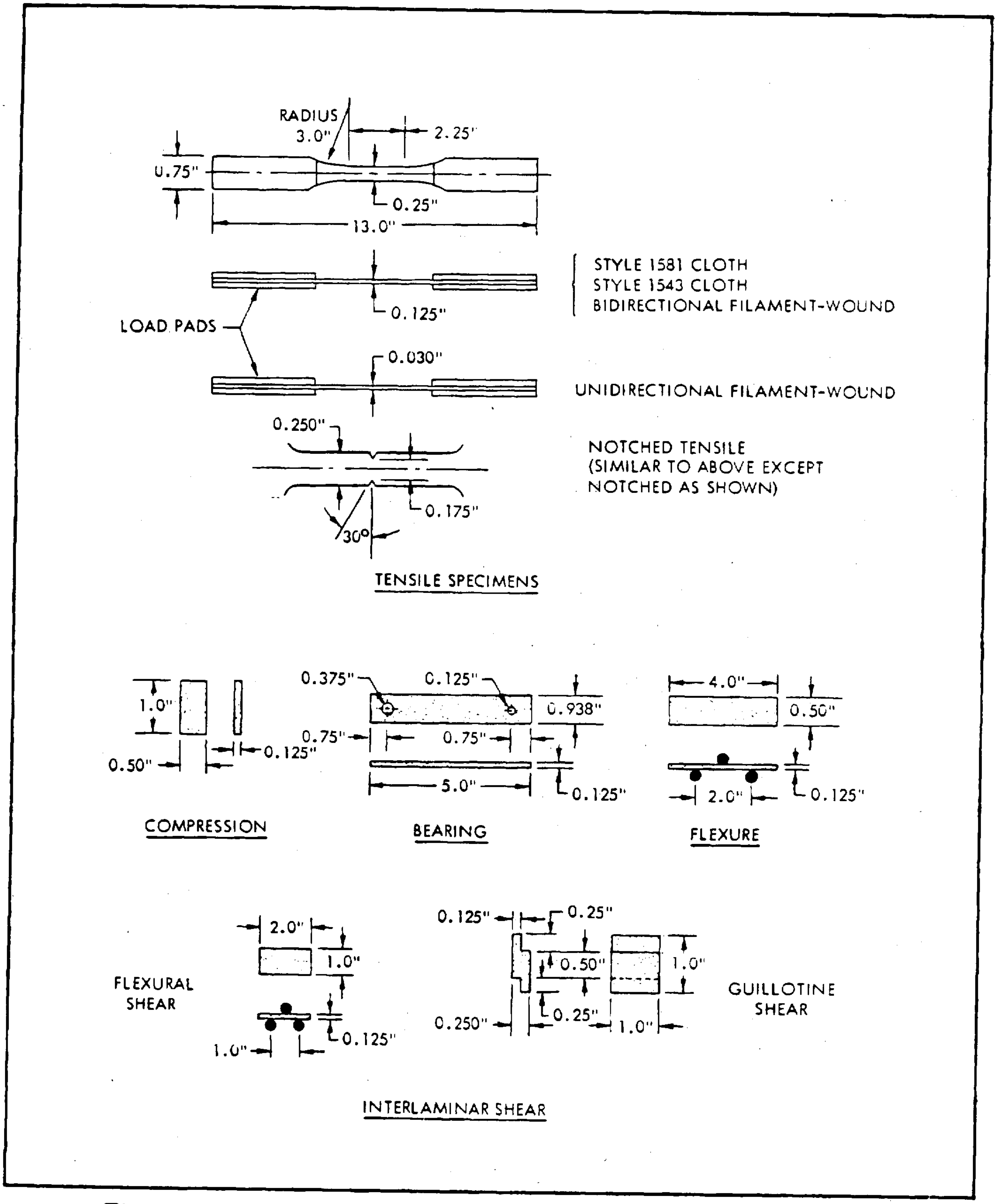

Figure 1 - Specimens for the Mechanical Properties Test Program

Structural Models

The purpose of the structural models was to determine the validity of the test data derived from the small test specimens when related to actual structural shapes. However, these structures also had to be relatively small to be tested at cryogenic temperatures in the cryostats available. Therefore, it was decided that the structural models would be rods and tubes as shown in Figures 2 and 3. These models were tested in tension, compression, and flexure.

Test Equipment and Procedures

General

The attainment of valid and useful mechanical and thermophysical properties data on engineering materials is dependent not only on the specimen configuration, but also on the standardization of test equipment, measurement techniques, and test procedures. Methods of testing plastic materials are contained in generally accepted standards such as ASTM and Federal Test Method No. 406. However, the unique circumstances involved in testing at cryogenic temperatures and the specific problems associated with testing the various reinforced plastic materials of this program made the direct utilization of these standards impractical.

Economically and technically feasible test methods and equipment were developed which have proved successful in the evaluation of reinforced plastic materials at cryogenic temperatures. Major factors that were considered included cryostat design, selection of instrumentation systems, and test fixture design.

Mechanical Properties Tests

General — The mechanical properties testing program included tension,

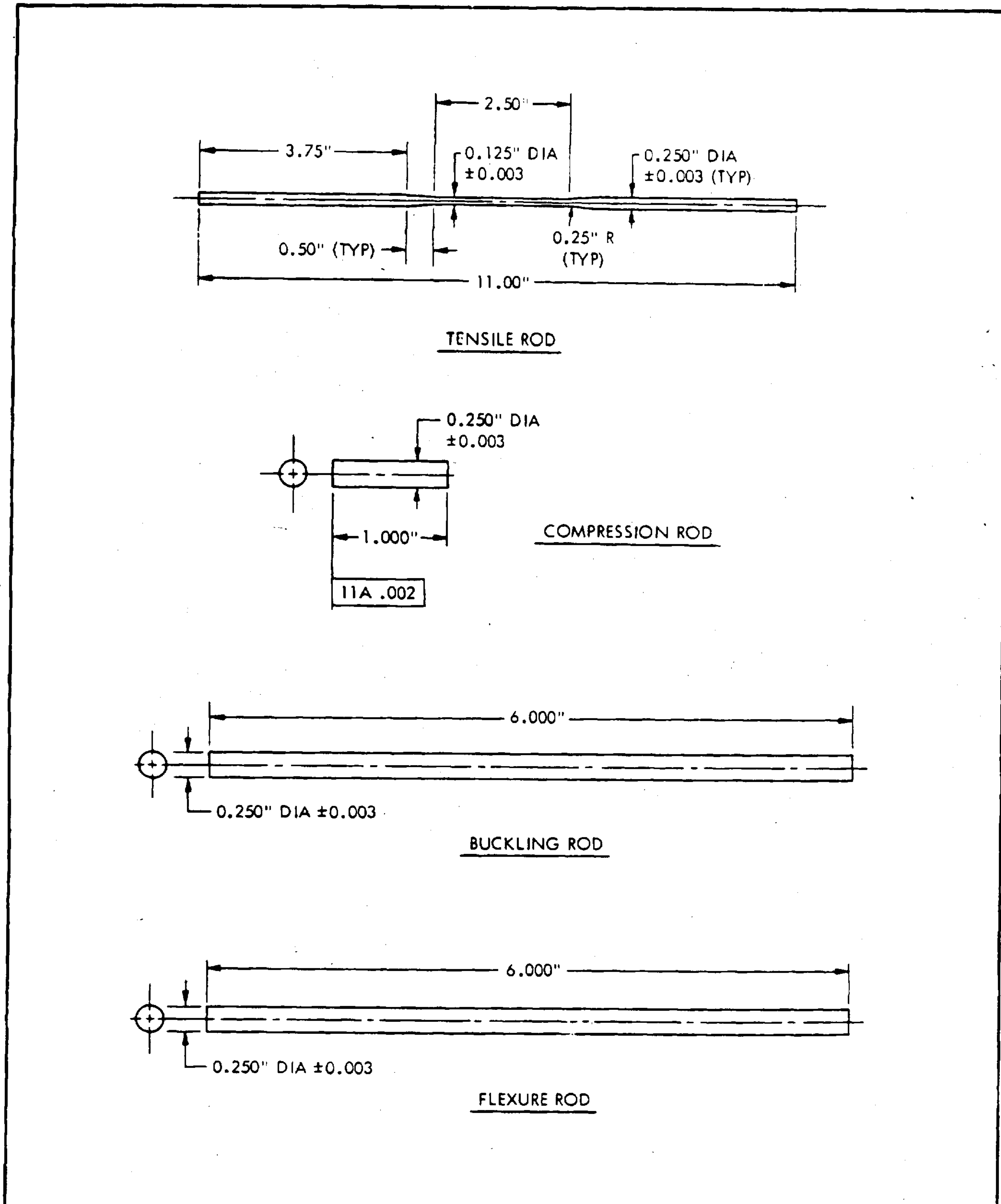

Figure 2 - Dimensional Sketches of Rod Test Specimens

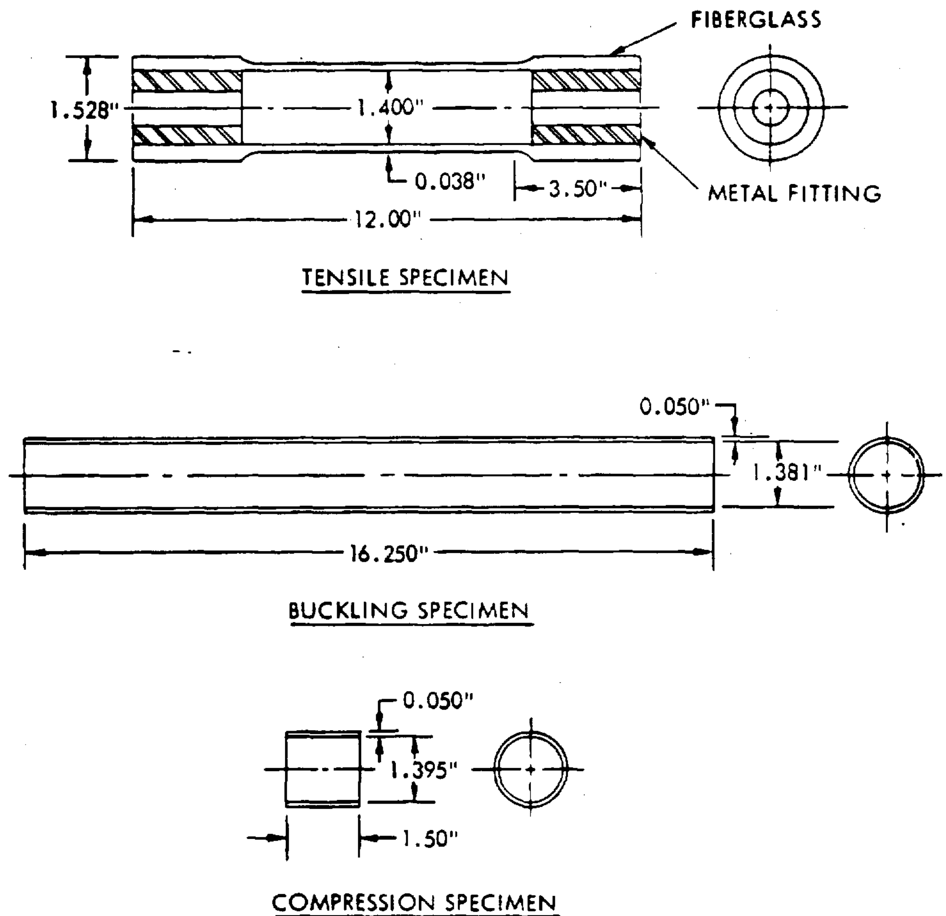

Figure 3 - Dimensional Sketches of Tube Test Specimens

compression, flexure, shear, and bearing tests. The rate of loading for each test was selected so that failure of the test specimen would occur within a time span of two to three minutes. The rate of load application was held uniform during each test. By conducting tests using time under load as the criterion for establishing cross-head travel or load rate, results of tests become independent of type of testing machine, design of cryostat, or rigidity of pull rods.

Test Equipment — Test equipment included testing machines, controlled temperature chamber and cryostats, devices for temperature measurements, liquid level measurements, and strain measurements, and test fixtures.

Universal testing machines utilized included a 60,000-lb capacity Baldwin, a 120,000-lb capacity Baldwin, and a 10-000-lb capacity Instron. Each machin is provided with several full-scale ranges and thus enables proper selection of ranges for the wide variation in the ultimate loads due to type of material or test.

Test temperatures of 197, 77, and 20 K were obtained by a controlled temperature chamber, a liquid nitrogen cryostat, and two liquid helium cryostats respectively.

The controlled temperature chamber, shown in Figure 4, is a circulating air-type chamber and provides test temperatures down to 90 K, using liquid nitrogen as the refrigerant. The temperature in the chamber is sensed by thermocouples and is automatically controlled to within ±1 K. Specimens were allowed a minimum of 30-min exposure at 197 K prior to testing.

The liquid nitrogen cryostat is shown schematically in Figure 5. The cryostat consists of a stainless steel compression tube, end plate, pull rod,

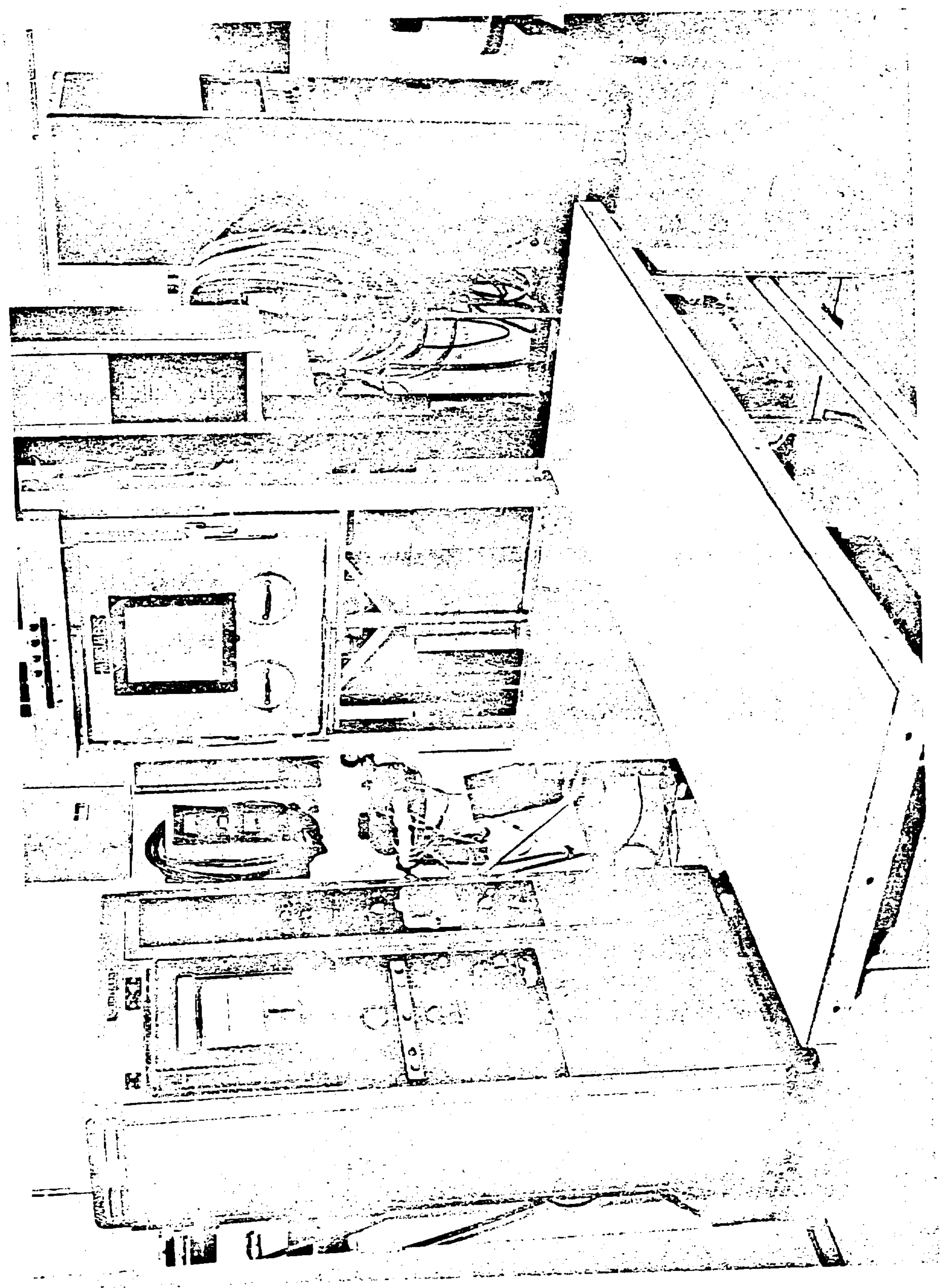

Figure 4 - Controlled Temperature Chamber Installed in Instron Testing Machine

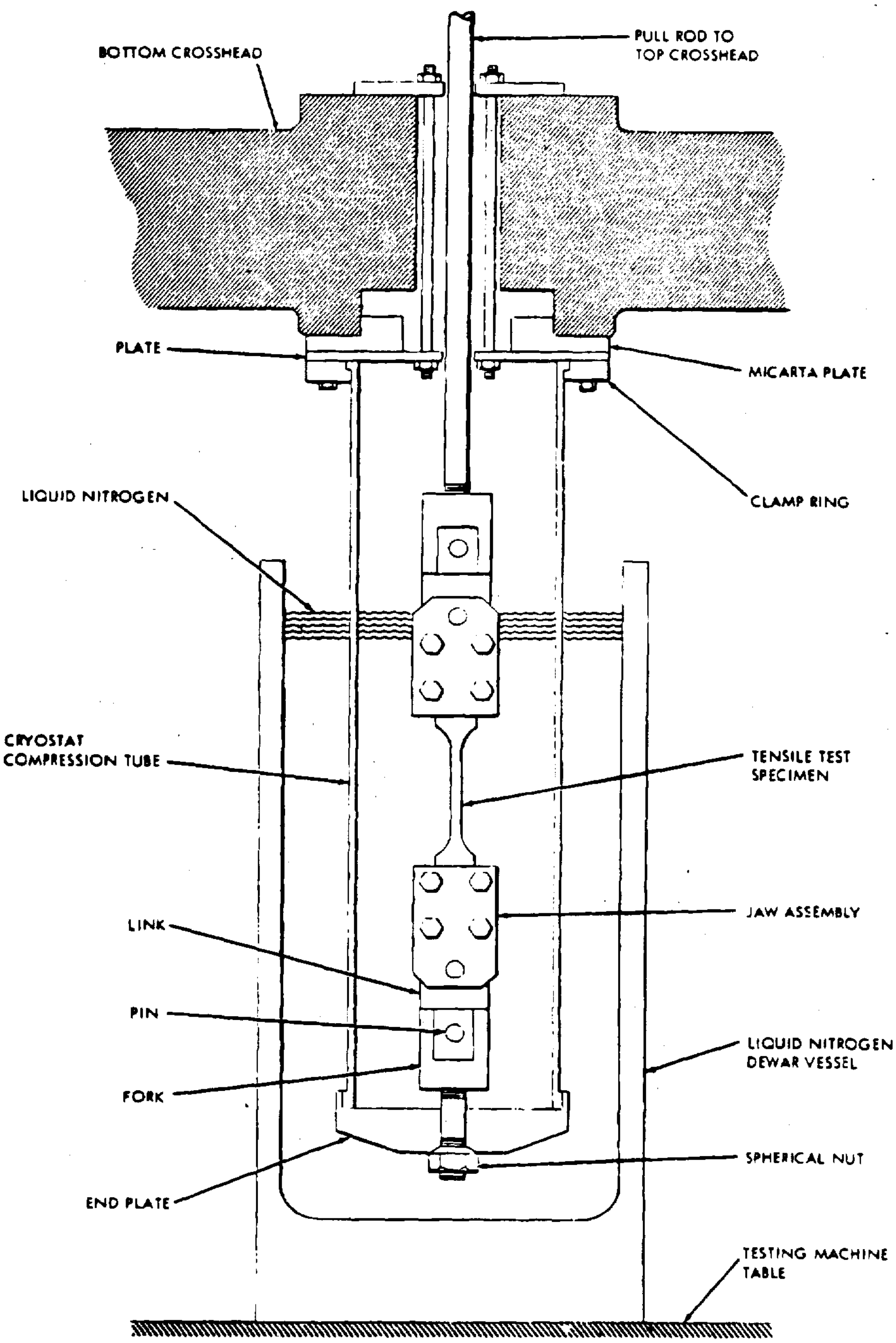

Figure 5 - Liquid Nitrogen Cryostat Assembly

and attachment devices for transmitting forces of up to 30,000 lb to the test specimen from the testing machine crosshead. The compression tube and end plate are perforated to permit liquid nitrogen to enter inside the tube and surround the test specimen. Liquid nitrogen is contained in a commercially available, stainless steel, open-mouth Dewar. The compression tube and the assembled test specimen are submerged in the liquid nitrogen until boiling of the nitrogen has ceased, thus assuring that the specimen is at 77 K.

Two liquid helium/hydrogen tensile cryostats were used in performing the 20 K mechanical properties tests. Both cryostats are constructed as shown schematically in Figure 6. One of the cryostats is capable of transmitting a force of 5000 lb and the second cryostat has a pull capacity of 15,000 lb. The cryostats, shown installed in testing machines in Figures 7, and 8, are of the internal compression-tube type construction. All components of the cryostats are fabricated of 300 series stainless steel except the pull rods and compression tubes, which are a titanium alloy. The cryostats are vacuum insulated and liquid nitrogen shielded. Each cryostat is provided with a three-point liquid level probe, a platinum resistance thermometer, and sufficient lead wires for use of extensometers, thermocouples, and heaters. In conducting tests at 20 K, the test chamber was first cooled to approximately 80 K with liquid nitrogen before liquid helium transfer was initiated. Temperatures were controlled by manually regulating the flow of liquid helium into the cryostat. An electric disk heater enabled quick vaporization of any collection of liquefied gases at the bottom of the test chamber.

Measurements — Temperature, liquid level and strain measurements were made. Commercially available platinum resistance thermometers and premium grade copper-constantan thermocouples were used for temperature

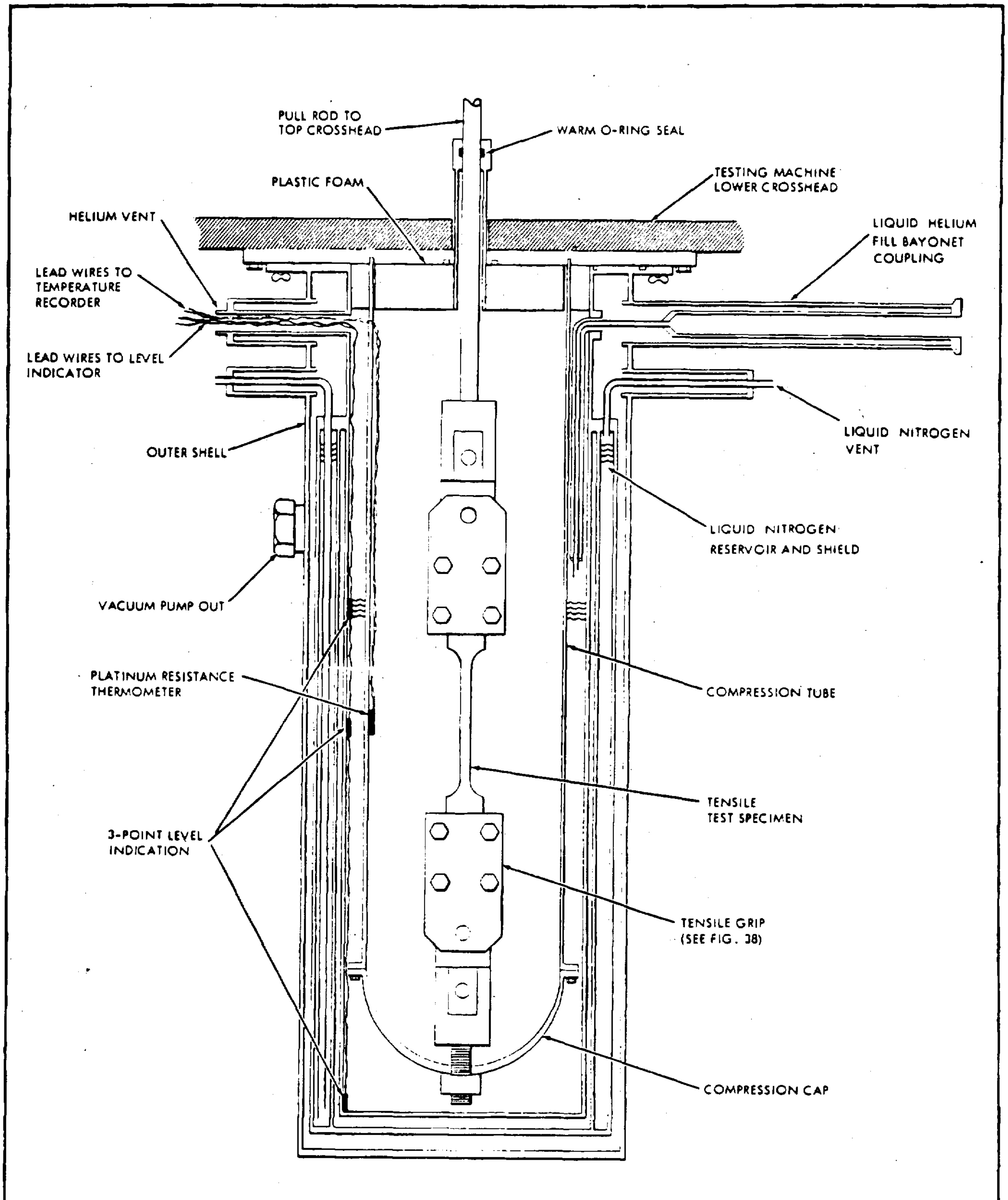

Figure 6 - Liquid Helium/Hydrogen Cryostat

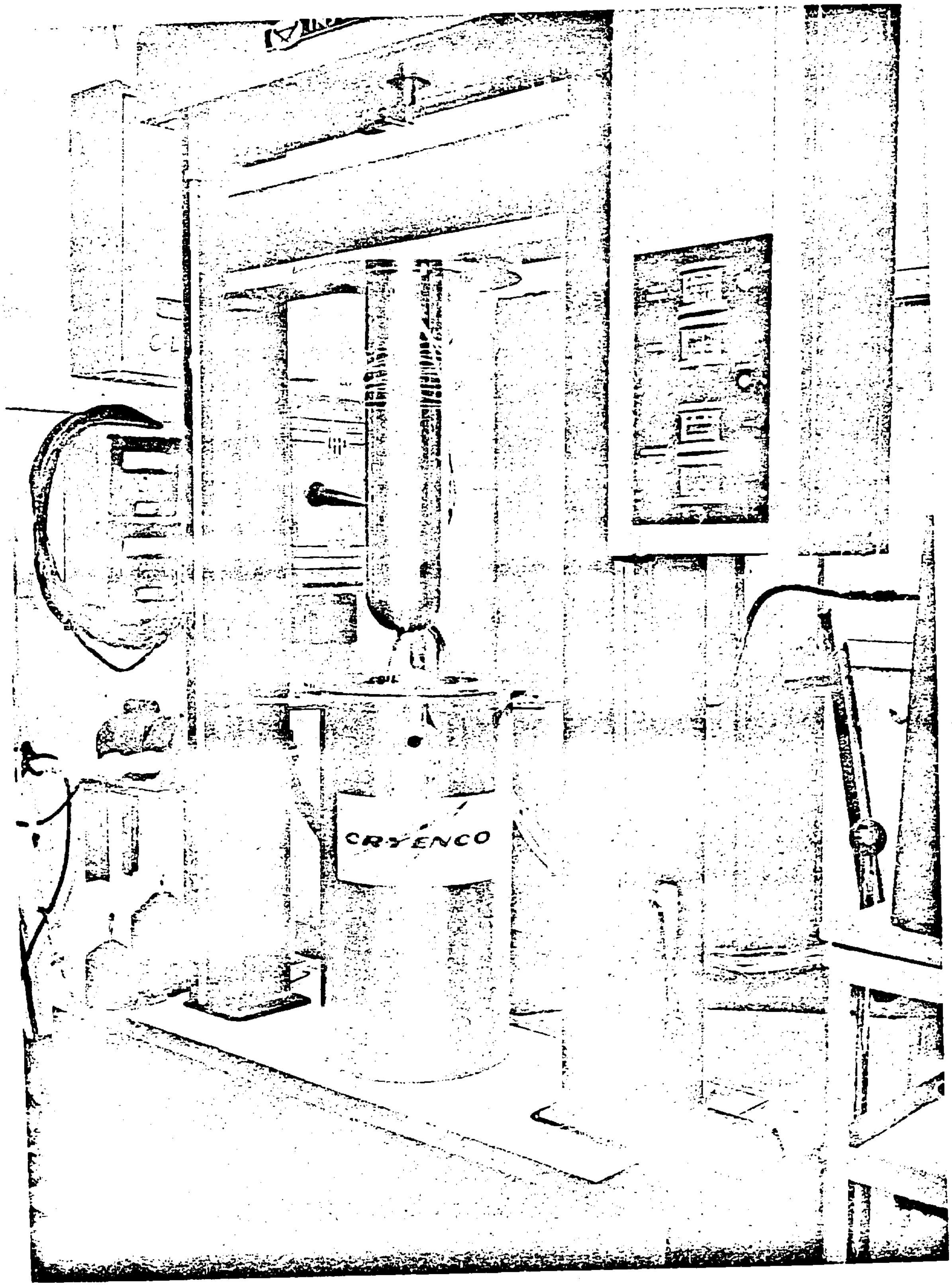

Figure 7 - 5000-Lb Capacity Liquid Helium Cryostat with Compression Tube Assembly Removed

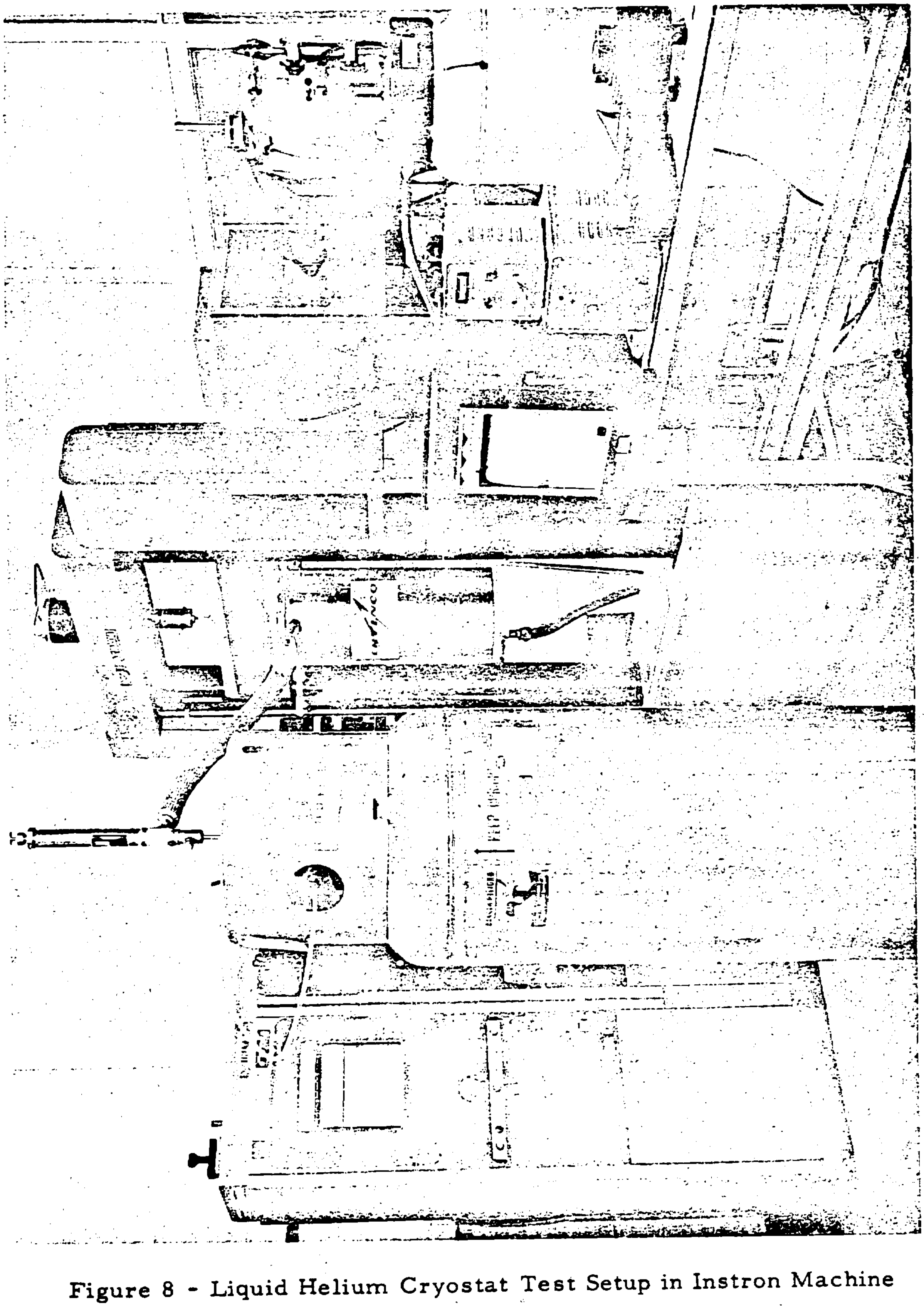

Figure 8 - Liquid Helium Cryostat Test Setup in Instron Machine

measurements in the range from 15 to 300 K. Each platinum thermometer was operated with a regulated d-c power supply and triple-bridge unit so that changes in temperature of the sensor were proportional to the measured voltage output from the triple bridge. Voltage measurements associated with either the platinum thermometer sensor systems or the thermocouples were made with precision potentiometers and/or potentiometric-type strip chart recorders.

Liquid nitrogen and helium levels in the liquid helium cryostats were measured with commercially available, carbon-resistor-type level indicators. The resistors were operated in a bridge circuit so that changes in resistance which resulted when the sensors were immersed in liquid nitrogen or helium could be measured on a panel-type microampere meter.

Mechanical properties of tensile modulus, bearing strength, and tensile hysteresis required the use of a strain-measuring device capable of operation at temperatures down to 20 K. The instrument selected for this task was a commercially available LVDT-type extensometer modified slightly for use at cryogenic temperatures. Since mechanical motion in the extensometer consisted of one tube sliding inside another, an inner tube was fabricated from copper so that binding between it and the stainless steel outer tube was eliminated when the extensometer was exposed to reduced temperatures. Lead wires to the extensometer were insulated with thin Teflon sleeves.

Special test fixtures were designed and fabricated for use in each type of mechanical properties test. Design parameters for all fixtures included proper specimen alignment, minimizing of heat leak into the cryostat, minimizing heat sink, and capability of operation in relatively small, inaccessible test space. Description of the test fixtures is included in the following discussion of test procedures.

properties of fiberglass-reinforced plastics is the design of a method for gripping the test sample without (1) jaw slippage or (2) inducing premature failure of the test sample at the jaws. When tests are conducted at cryogenic temperatures, the problem is complicated further because of (1) limited space for grips, (2) relative inaccessibility of the gripping system, and (3) excessive heat sink of conventional grips.

After preliminary testing, involving various concepts in specimen configuration and fixture design, a system was developed which enabled the successful testing of all materials at all temperatures. The special jaws shown in Figure 9 were fabricated and used in tests without either jaw slippage or premature failure of the test sample outside of the test area. The alignment fixture shown in Figures 10 and 11 was fabricated and used to provide good alignment of the specimen in the jaws. No evidence of premature specimen failure due to specimen misalignment was encountered.

A typical tensile test setup is shown inFigure 12. The extensometer is a commercially available LVDT type with little modification. It is spring-mounted to the specimen. Two small plastic plates are placed on the back side of the specimen to aid in holding the knife edges of the extensometer firmly against the specimen. Holes are drilled in the plastic plates, and a steel rod is inserted to tie the plates together. The holes are drilled large enough to prevent binding of the rod. By using this method to mount the extensometer, the center section of the test specimen remains straight and counterbalancing of the extensometer is unnecessary.

Elongation of the test specimen was sensed by the extensometer and autographically recorded by the testing machine recorder. The graph of specimen

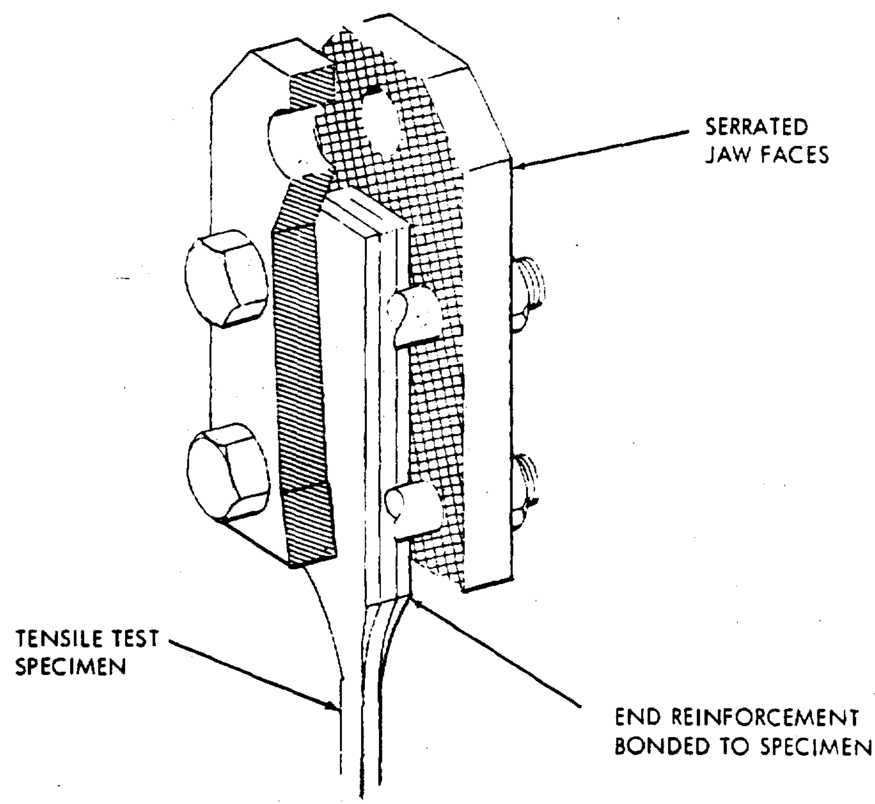

Figure 9 - Tensile Test Jaw Assembly

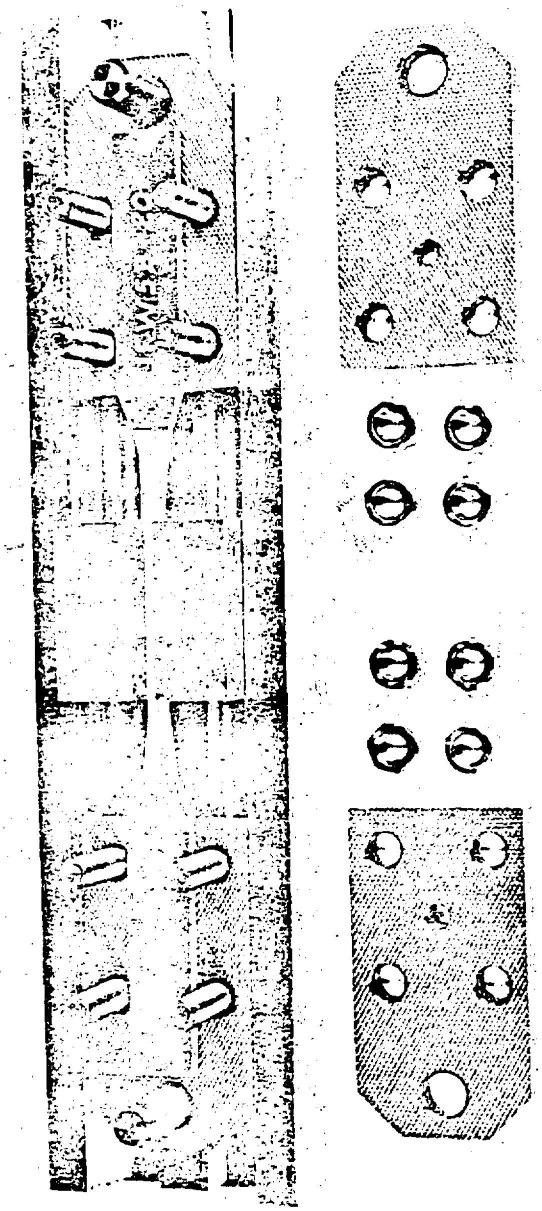

Figure 10 - Alignment Fixture, Jaws, and Tensile Specimen

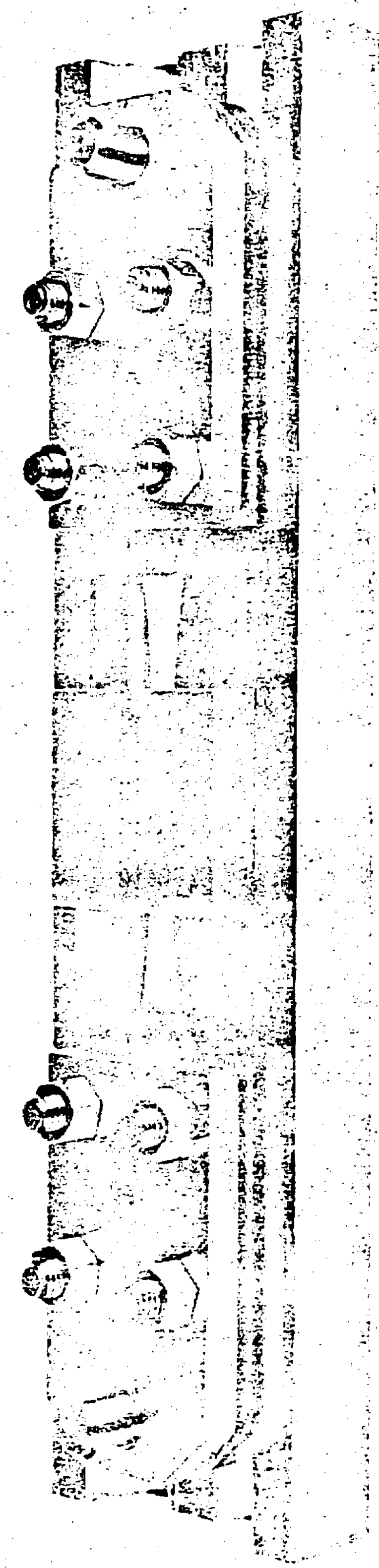

Figure 11 - Assembly of Tensile Specimen with Jaws in Alignment Fixture

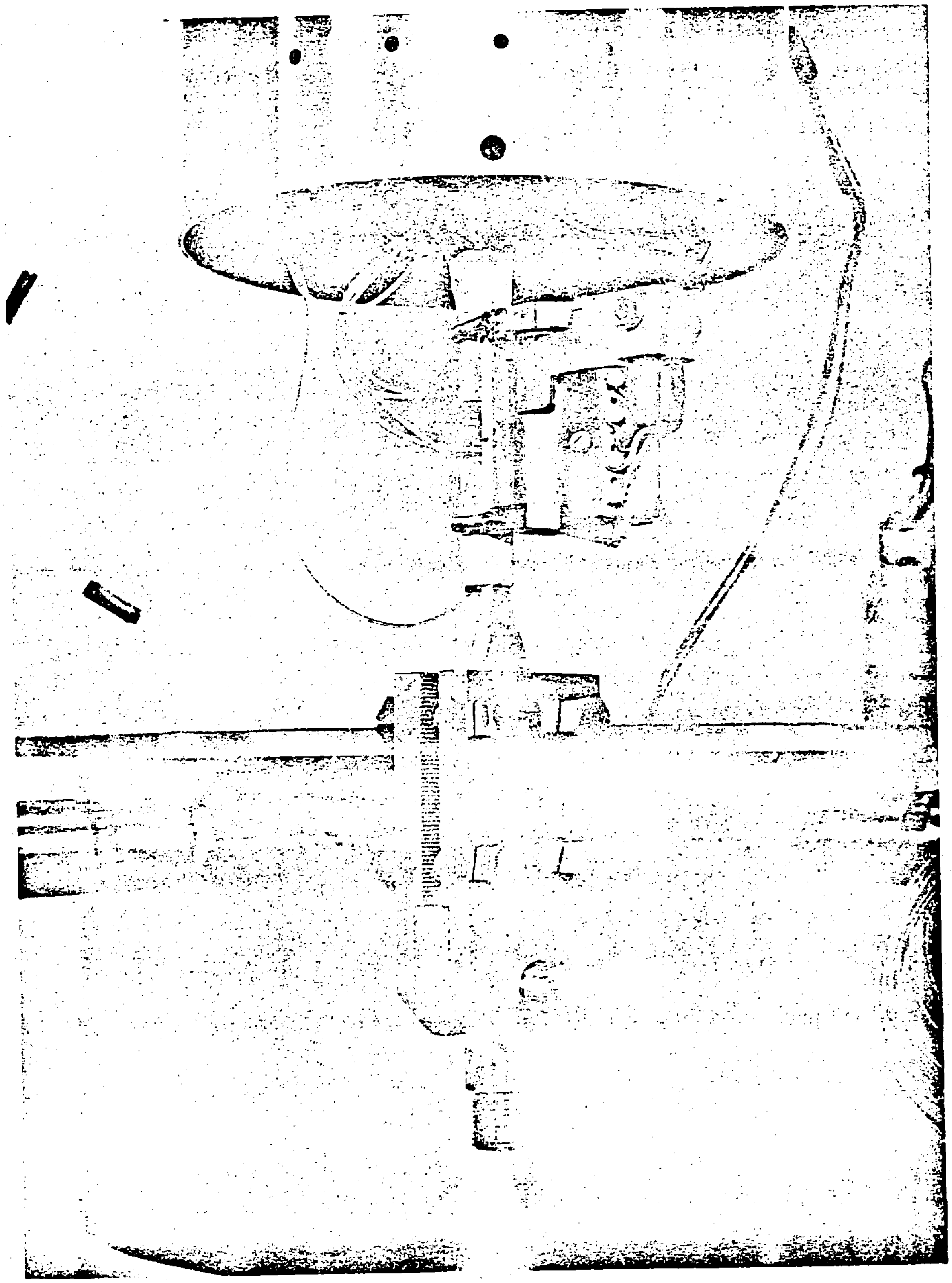

Figure 12 - Tensile Specimen with Extensometer being Installed in Compression Tube of Cryostat

elongation versus load was used to determine Young's modulus and percent elongation of the specimen.

Notched Tension Test Procedures — Notched tension tests were performed using the same equipment and procedures as those described for the unnotched tension specimens except that no extensometer was required. These tests yielded ultimate tensile strengths needed to obtain the notched/unnotched tensile strength ratio of each material.

Compression Test Procedures — Compression tests were conducted using the compression cage fixture shown in Figure 13. This fixture provides a means of transmitting forces from the testing machine crossheads to the specimen in the cryostat using the same pull rods as those used for tension tests. The greatest advantage in using this concept is that the load-carrying members of the fixture are subjected only to tensile stresses and therefore can be designed for a minimum heat path and heat sink.

An alignment fixture, Figure 14, was fabricated to enable accurate assembly of the end support blocks to each test specimen. The small bosses on each end of the assembled specimen mate with holes in the loading plates of the compression cage to permit precision alignment of test specimens in the compression cage test fixture inside the cryostats. The end support blocks were bonded to the test specimen with a room temperature curing epoxy adhesive and prevented premature failures due to end crushing.

Flexure Test Procedures — A test fixture (Figure 15) was fabricated to provide a means of performing flexure tests in the tensile cryostats. The fixture consists of two yoke assemblies and provides three rollers for loading the test specimens as simple beams over a two-inch span. The rollers are recessed in the center portions to ensure proper alignment of the specimen during cool-down and testing.

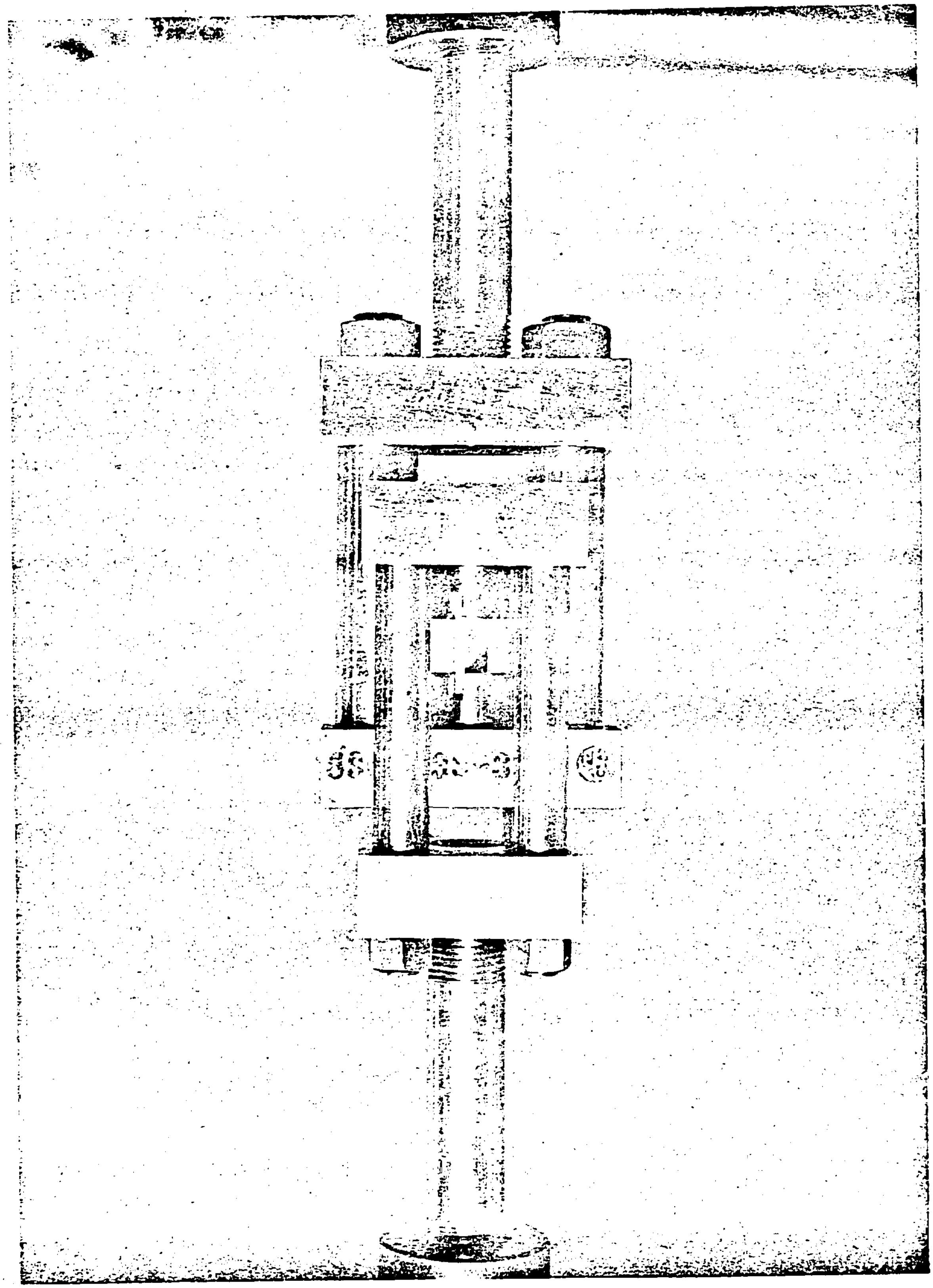

Figure 13 - Test Fixture for Conducting Compression Tests

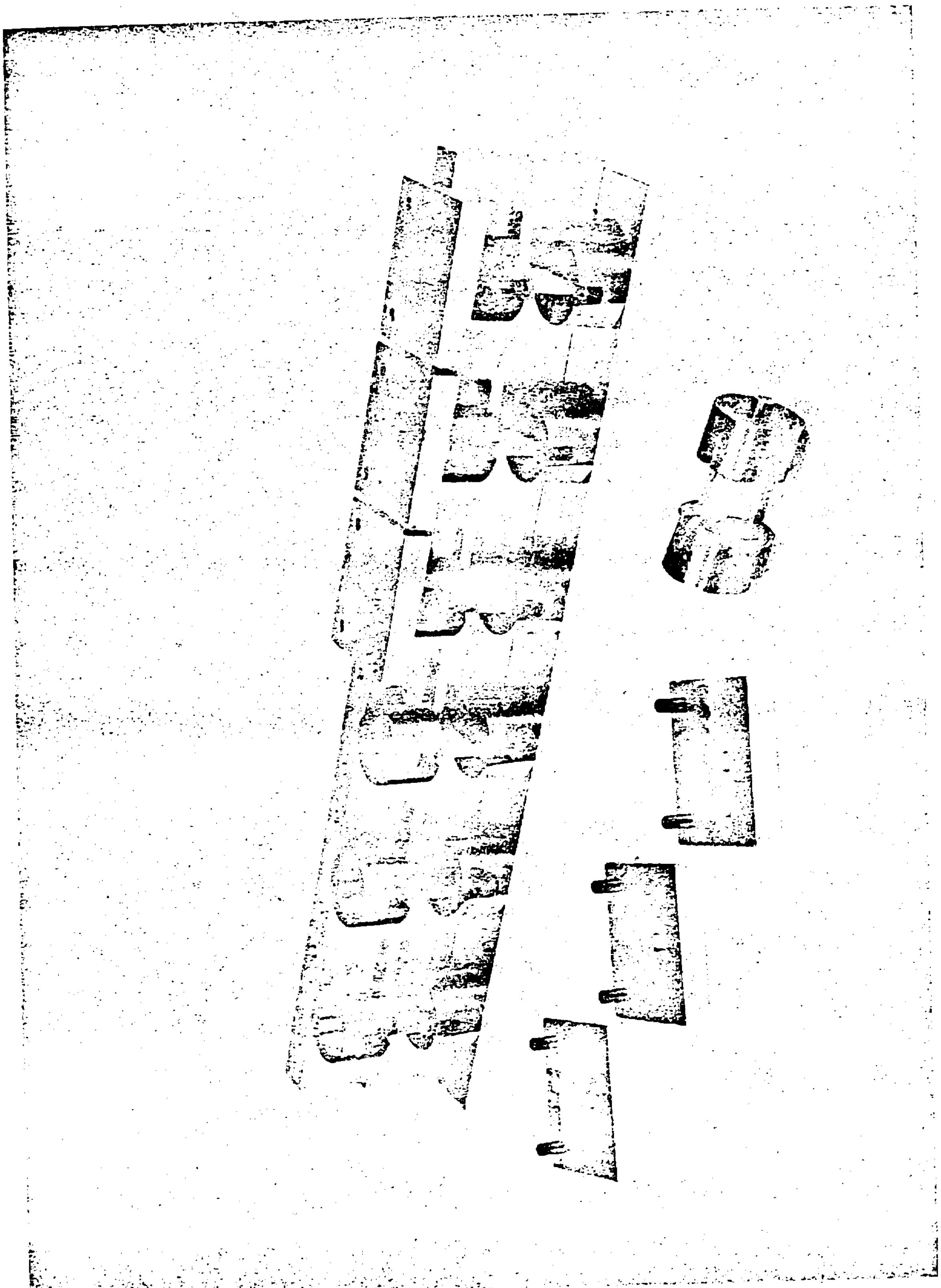

Figure 14 - Alignment Fixture for Bonding of Test Specimen to Edge Supports

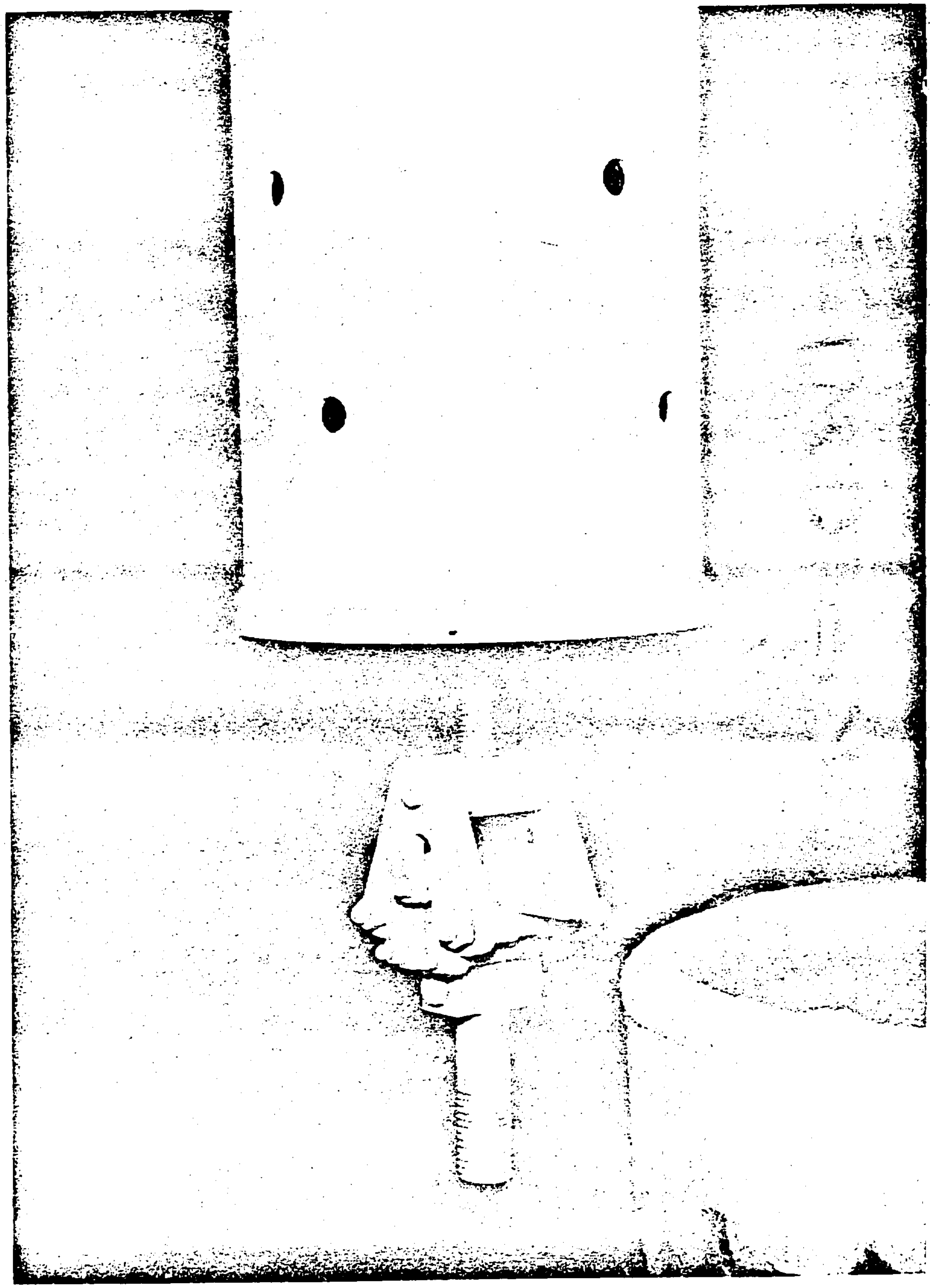

Figure 15 - Flexure Test Specimen and Fixture after Removal from Liquid Nitrogen Cryostat

The flexural moduli of the materials under study were obtained through the Instron machine, which automatically records head travel versus load. This information was used to compute flexural modulus. However, the recorded deflection includes a small amount of deflection in the linkage of the machine itself, the cryostat tube, the test fixture, and the pull rods. A calibration test was performed at each test temperature to determine the amount of correction to be applied to the deflection as indicated on the recorder. The calibration consisted of substituting a steel bar 1/2 inch wide × 5/8 inch high × 4 inches long in place of the 1/8-inch thick plastic specimen and determining the overall deflection of the system. These data then were used to obtain the true load-deflection curve for each test specimen.

Bearing Test Procedures - Bearing strength tests were conducted using the fixture shown in Figure 16, which is in accordance with Method 1051 of Federal Test Standard No. 406. The test specimen was attached to one of the pull rods with a pin and clevis. The bearing force was applied to the specimen through a hardened steel pin. Deformation of the hole in the specimen was measured with the LVDT-type extensometer used in the tensile tests. Deformation versus load was recorded autographically on the Baldwin machine recorder.

Bearing yield stress was defined as the stress on the stress-deflection curve that corresponds to a deflection distance of four percent of the bearing hole diameter when measured from the intersection of a line tangent to the bearing stress-deflection curve at this point and the zero-load axis (see Figure 17). This method corresponds to the method outlined in Federal Test Method Standard No. 406, Method 1051; and since it is insensitive to zero-load errors, it improves the precision of the bearing strength measurement.

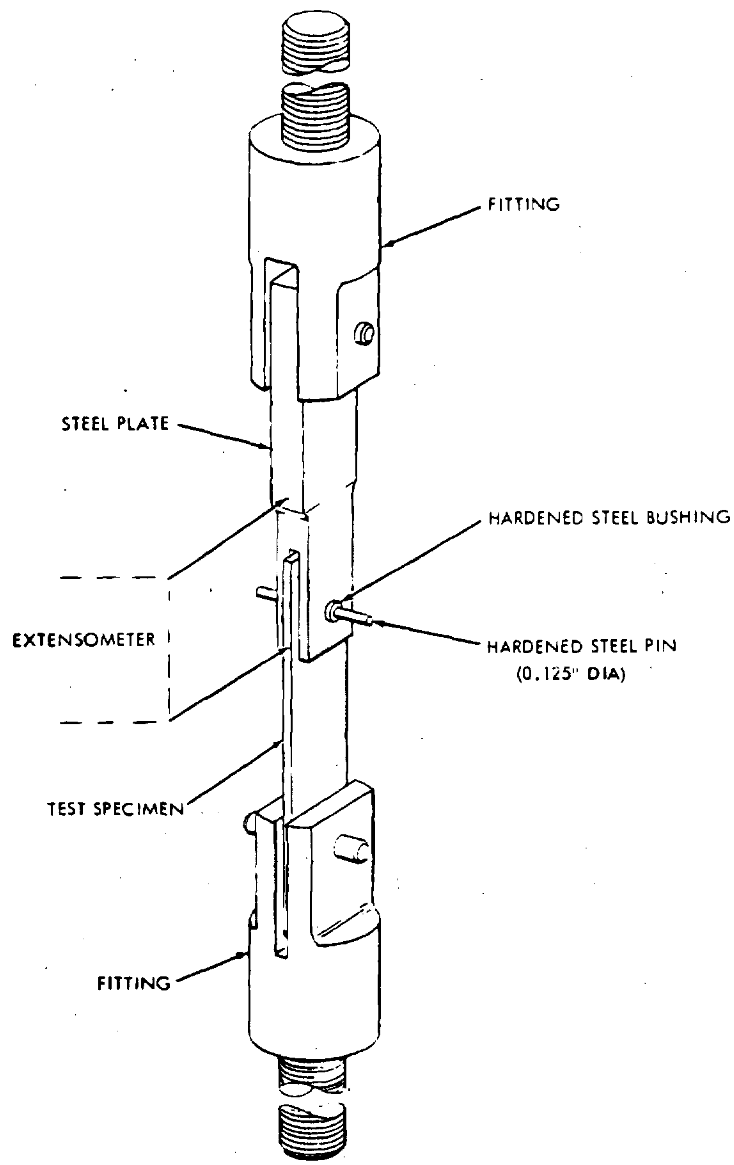

Figure 16 - Bearing Strength Test Fixture

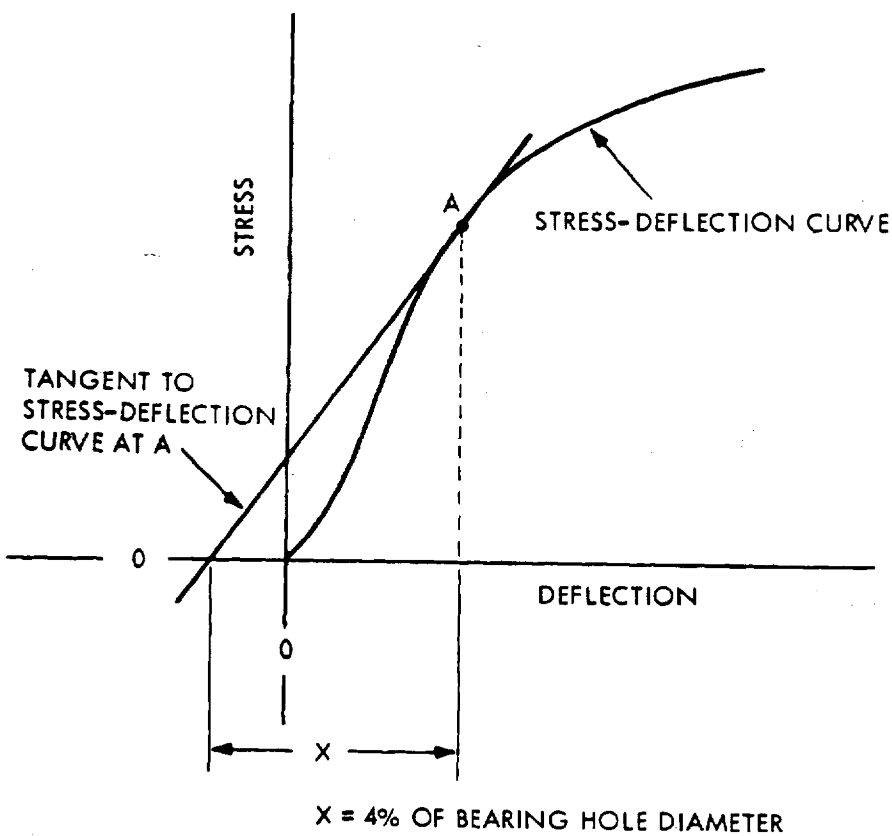

Figure 17 - Method of Determining Bearing Yield Strength from Bearing Stress-Deflection Curve

Interlaminar Shear Test Procedures - Two test methods were used to determine the interlaminar shear properties. Flexure shear tests were performed using the test fixture shown in Figure 18. The test fixture is essentially a short-span flexure device designed to induce interlaminar shear failure, rather than tensile or compressive failure of the outer fibers of the test specimen. The rollers in this fixture are recessed in the center portion, thus insuring proper specimen alignment during the test. The three rollers of the fixtures are located to test the specimen as a simple beam over a one-inch span. The guillotine shear tests were performed using the test fixture shown in Figure 19. The test fixture is placed in the compression gage and loaded in compression, with failure occurring in shear. This test requires a 1/4-in. thick specimen and careful machining, but provides less scatter and values that are more representative of design values.

Tension Hysteresis Test Procedures - Tension hysteresis tests were performed in the Instron Universal testing machine utilizing the same test fixtures and extensometer as those described for the tension tests. The testing machine is equipped with an "X-Y" chart drive system which is designed for use with standard LVDT-type extensometers. The system exhibits excellent stability and zero drift and is therefore very suitable for conducting hysteresis tests. Test specimens were automatically cycled by the machine between zero and a predetermined stress level.

Testing of Structural Models

Just as special test fixtures and methods were required for the mechanical properties testing program, the testing of the structural tubes and rods required that new test fixtures and methods be developed to transmit the expected loads. The sketches of the test fixtures fabricated to test the structural

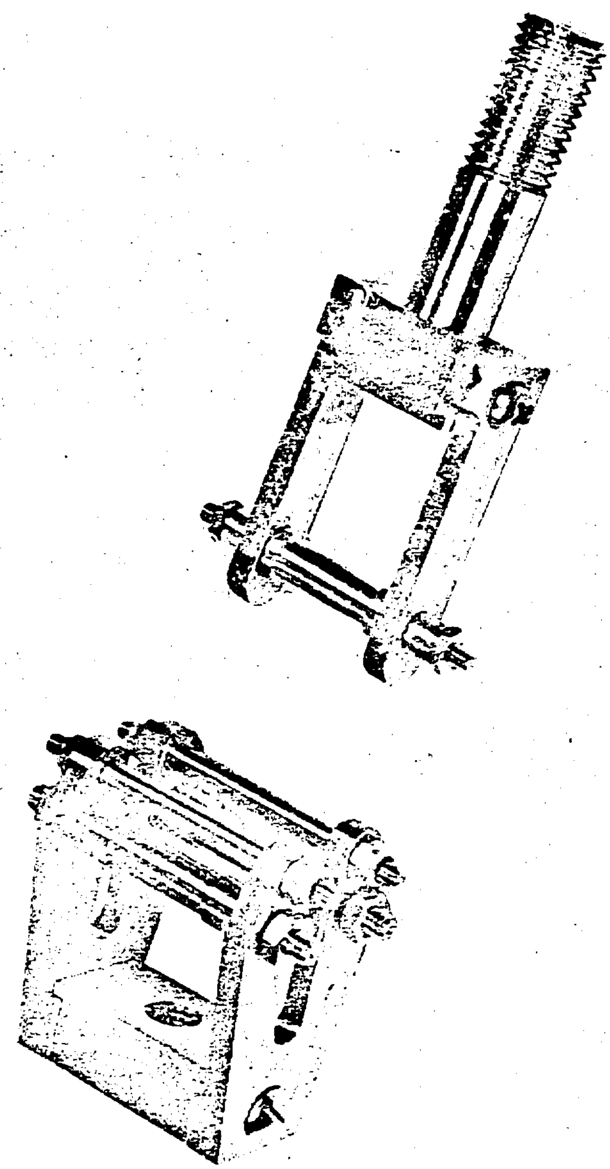

Figure 18 - Test Fixture for Interlaminar Shear Tests

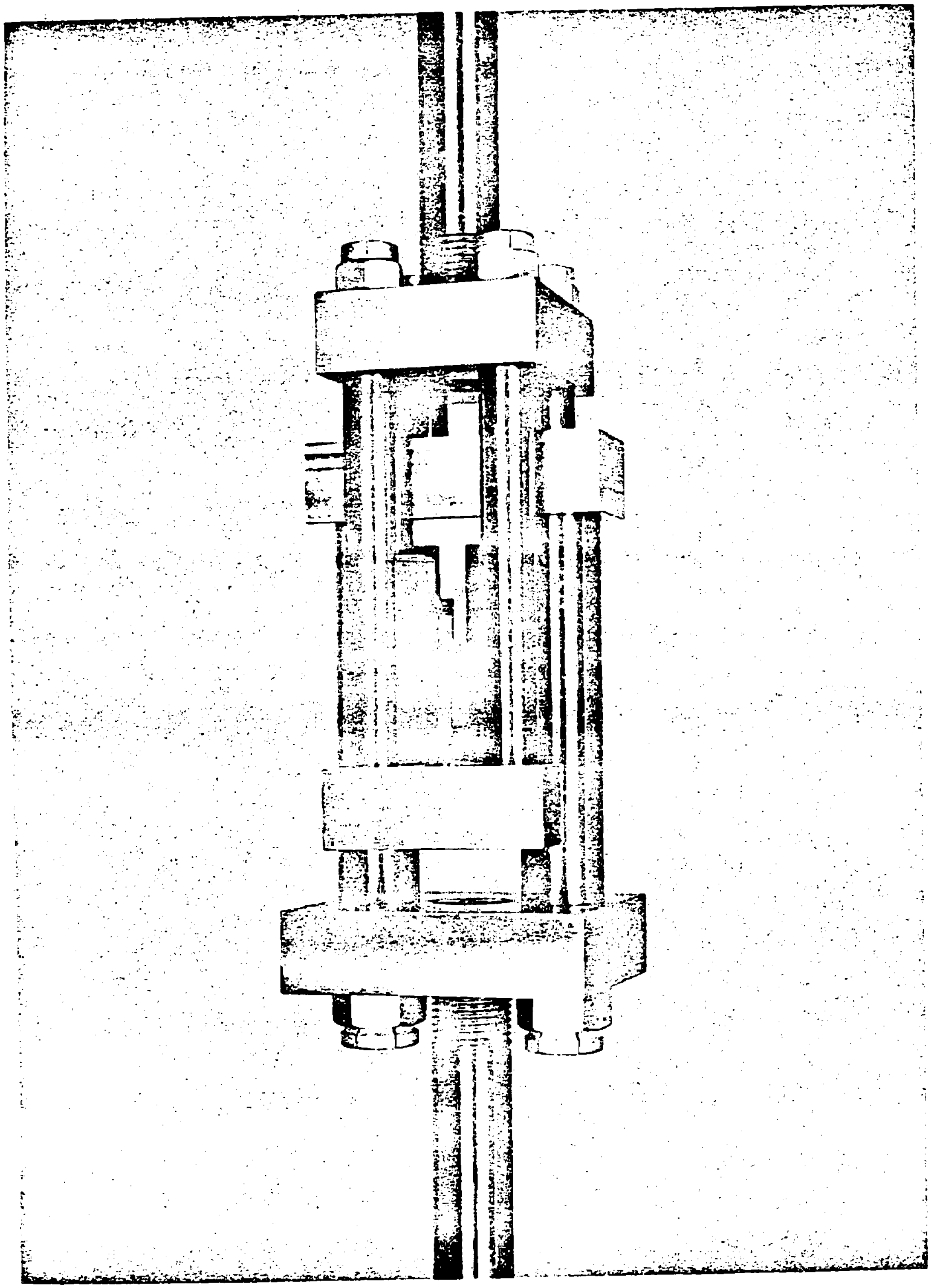

Figure 19 - Guillotine Shear Specimen in Compression Cage before Test

models in tension, compression, buckling, and flexure are shown in Figures 20 through 24. To enable the load to be transferred from these test fixtures into the specimens, special end fittings had to be bonded to the as-fabricated specimens. The steel fittings were acid etched and then the adiprene L-100 and mocha adhesive system used to bond in the specimens.

Preliminary tests were conducted on tensile tubes using only the internal loading plug. However, tension testing of this specimen produced a failure within the bond of the plug to the tube rather than a tensile failure of the tube. By adding the external sleeve bonded to the specimen, a failure within the specimen occurred.

Figure 25 shows the sleeves bonded to the tensile rods, and Figure 26 shows the assembly for the tensile tubes. The compression buckling specimens required end fittings that provide a socket for the load-transmitting metal balls. The fittings for the rod and tube specimens are shown in Figures 27 and 28. To provide proper alignment of the compression buckling specimens, special centering devices were fabricated and attached to the cryostat as shown in Figure 29. After a small initial preload is applied to the specimen, these centering rigs are removed for the actual test. The end fittings for the ultimate compression rod and tube specimens are shown in Figures 30 and 31. These specimens are sufficiently short that the ultimate compressive strength is reached before column buckling occurs; however, uniform loading is required so the end fittings must be maintained parallel to each other and perpendicular to the axial direction of the specimen. The test setup for the flexure test of the rod specimens is shown in the cryostat in Figure 32. The test setup after the addition of liquid nitrogen is shown

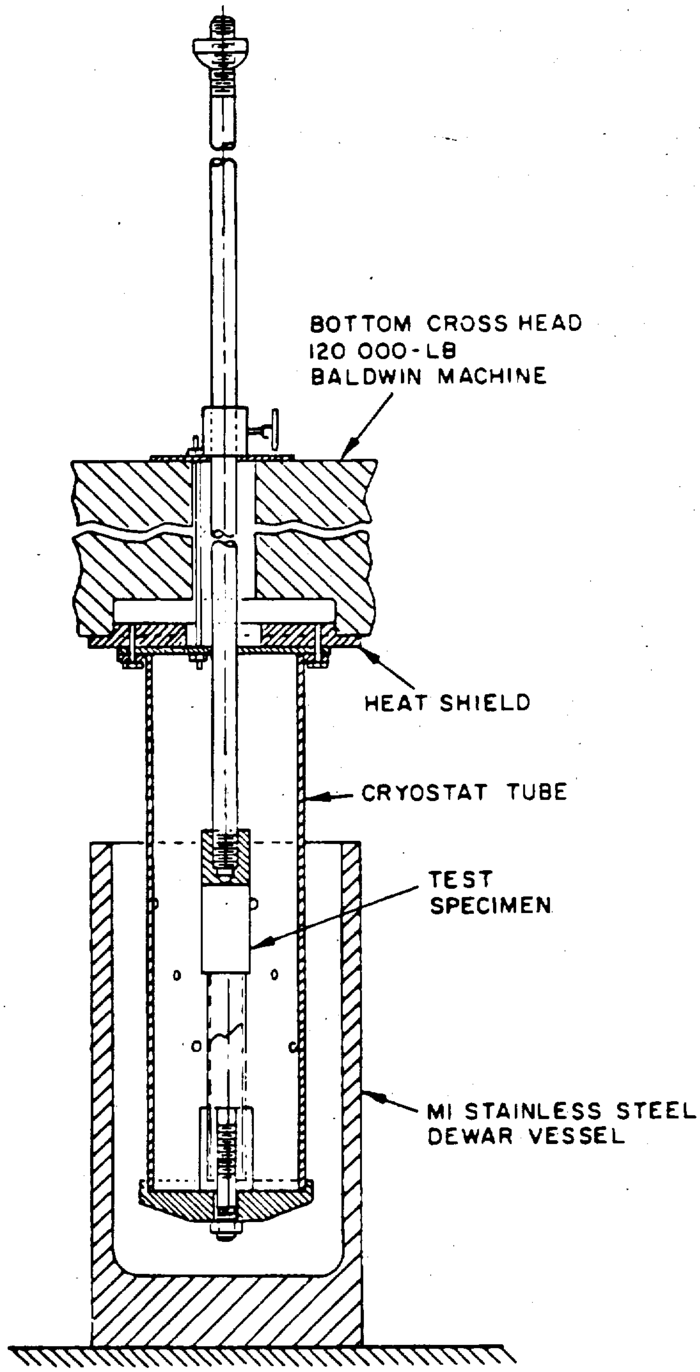

Figure 20 - Tensile Tube Test Setup

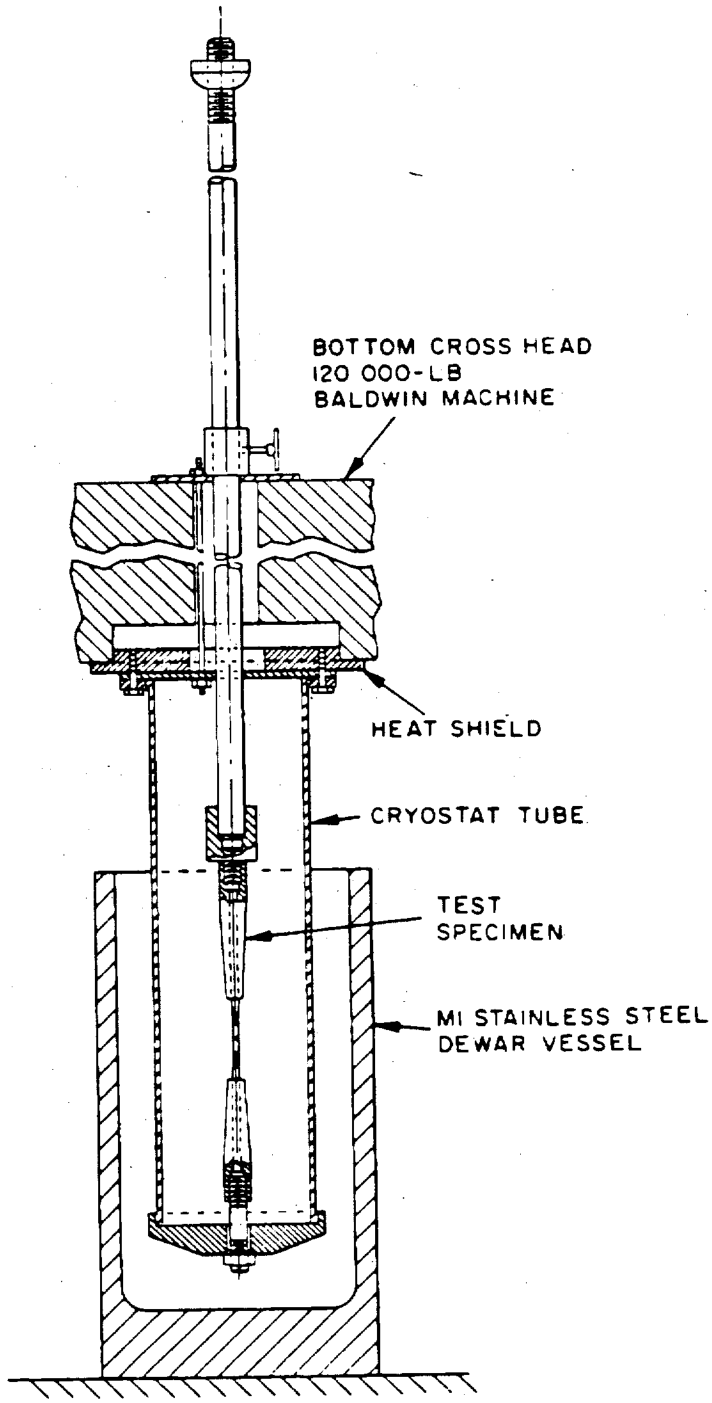

Figure 21 - Tensile Rod Test Setup

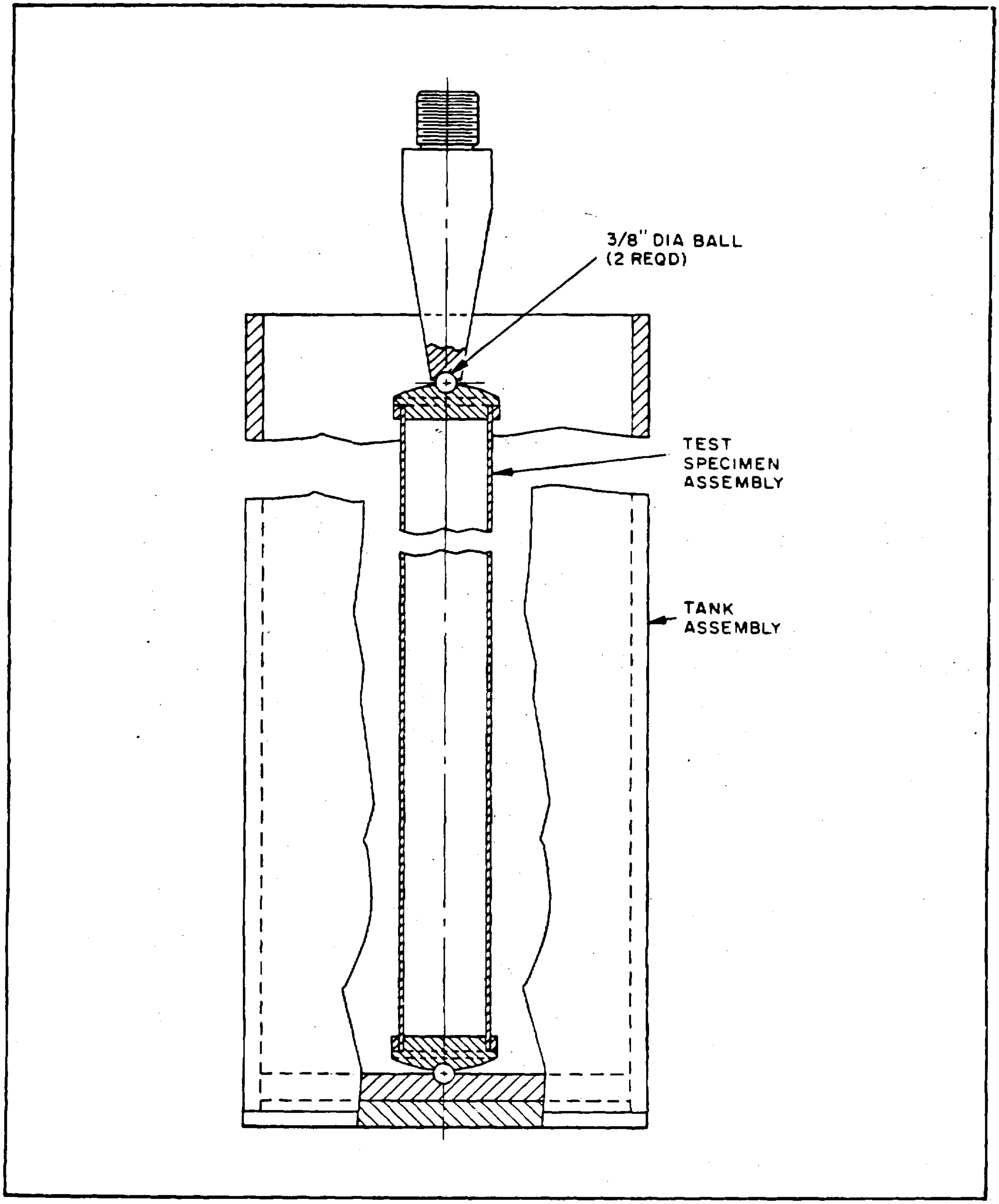

Figure 22 - Buckling Tube Test Setup

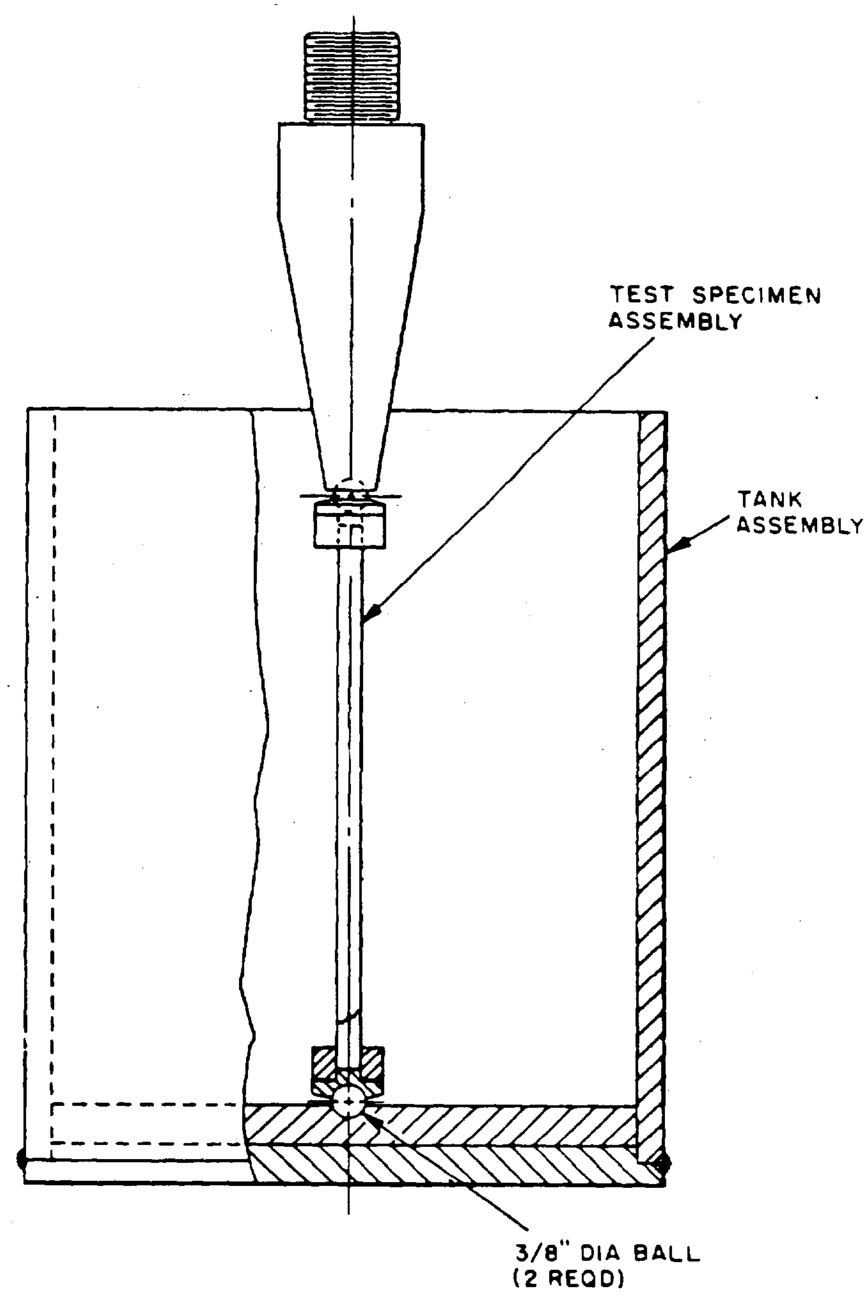

Figure 23 - Buckling Rod Test Setup

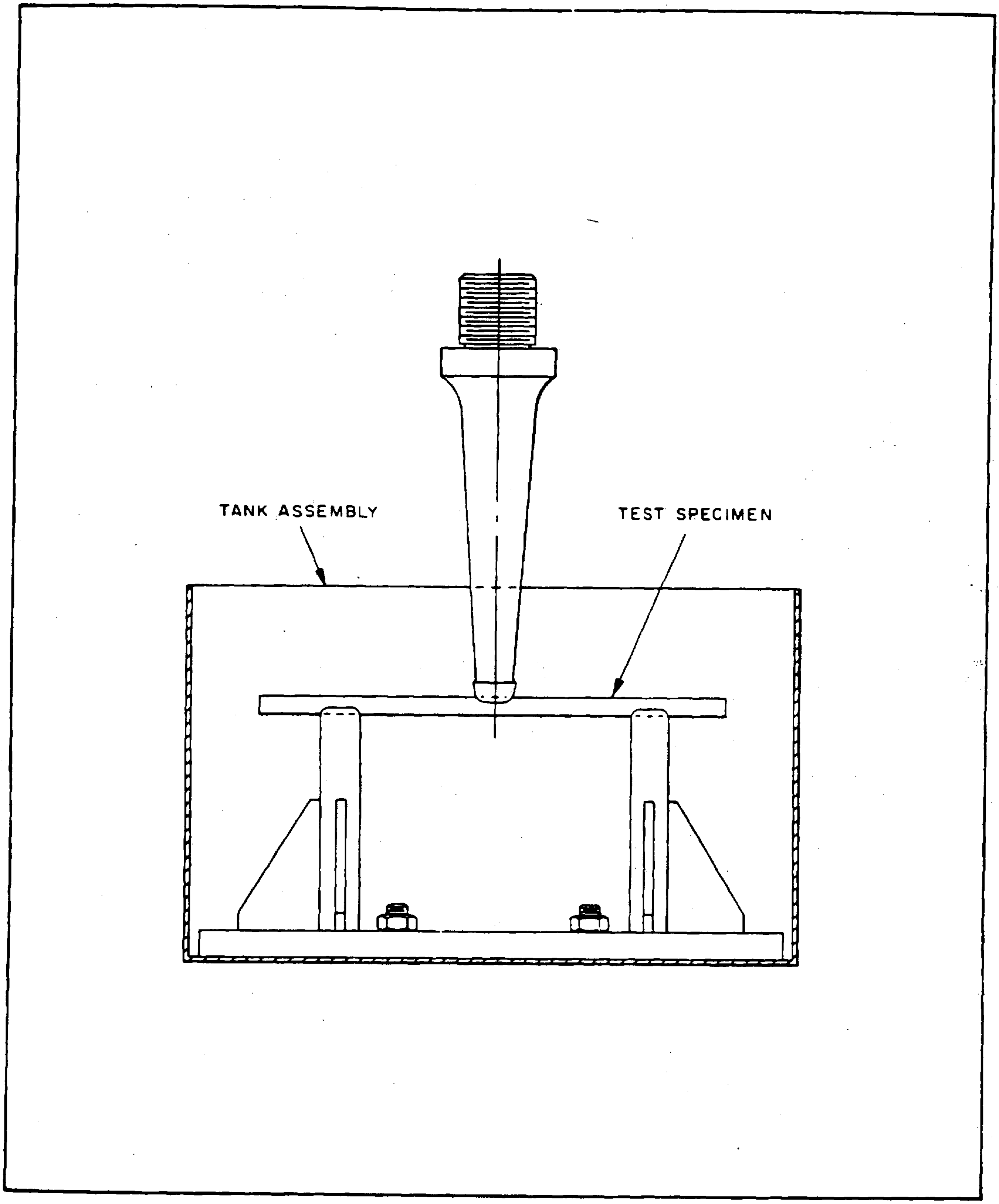

Figure 24 - Flexural Rod Test Setup

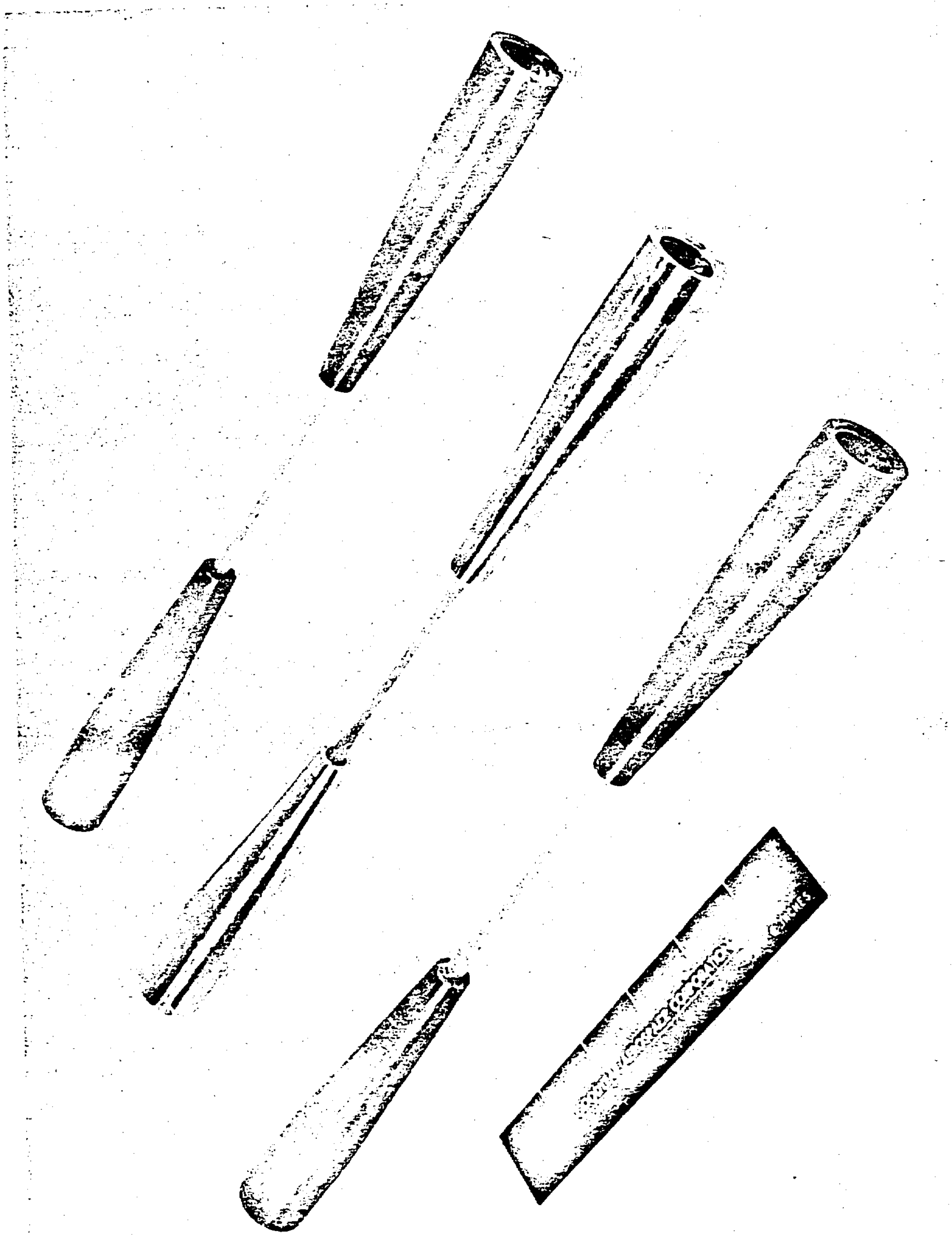

Figure 25 - UFW Tensile Rods before Test

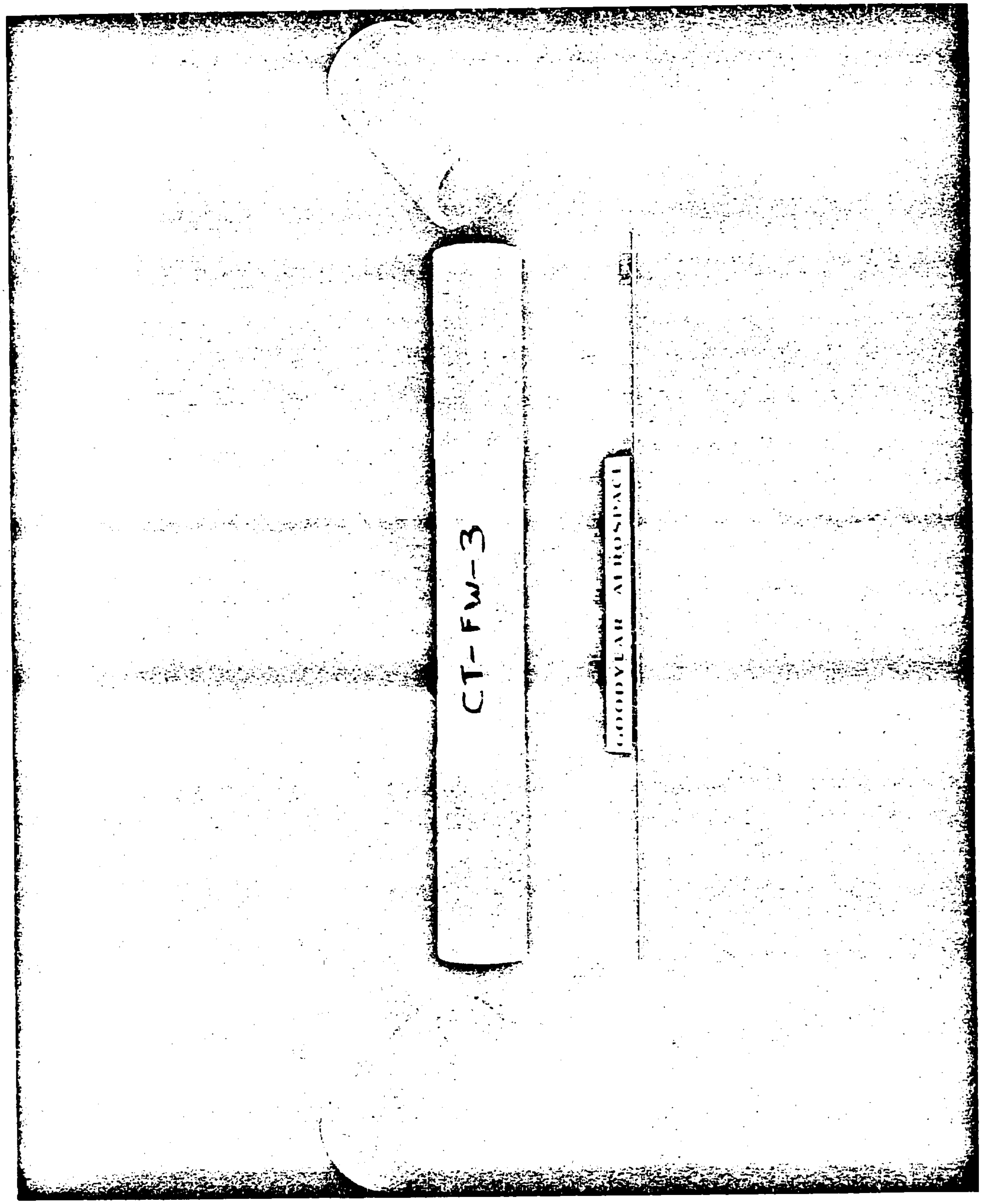

Figure 26 - Tensile Tube with Sleeves

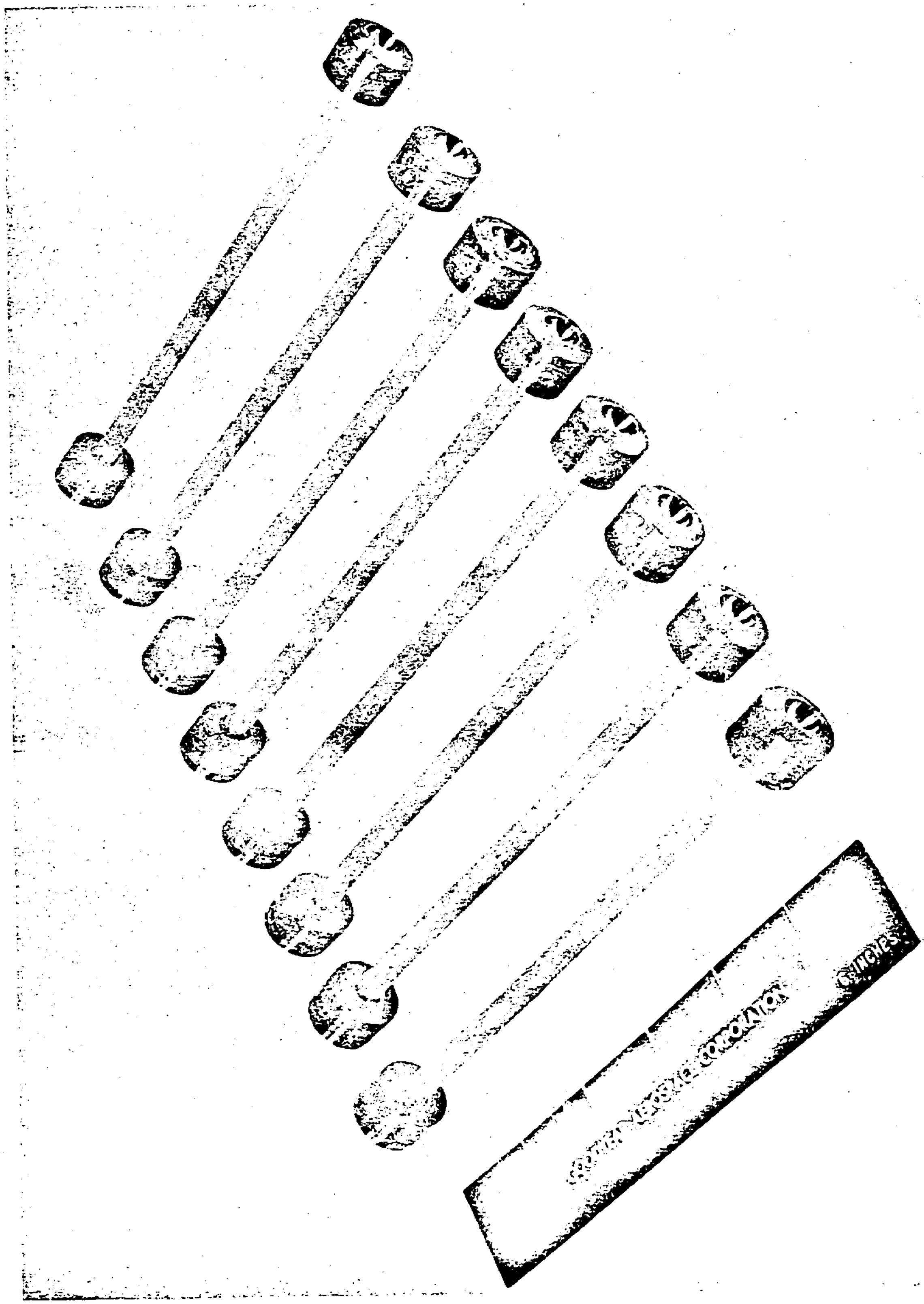

Figure 27 - Buckling Rods before Test

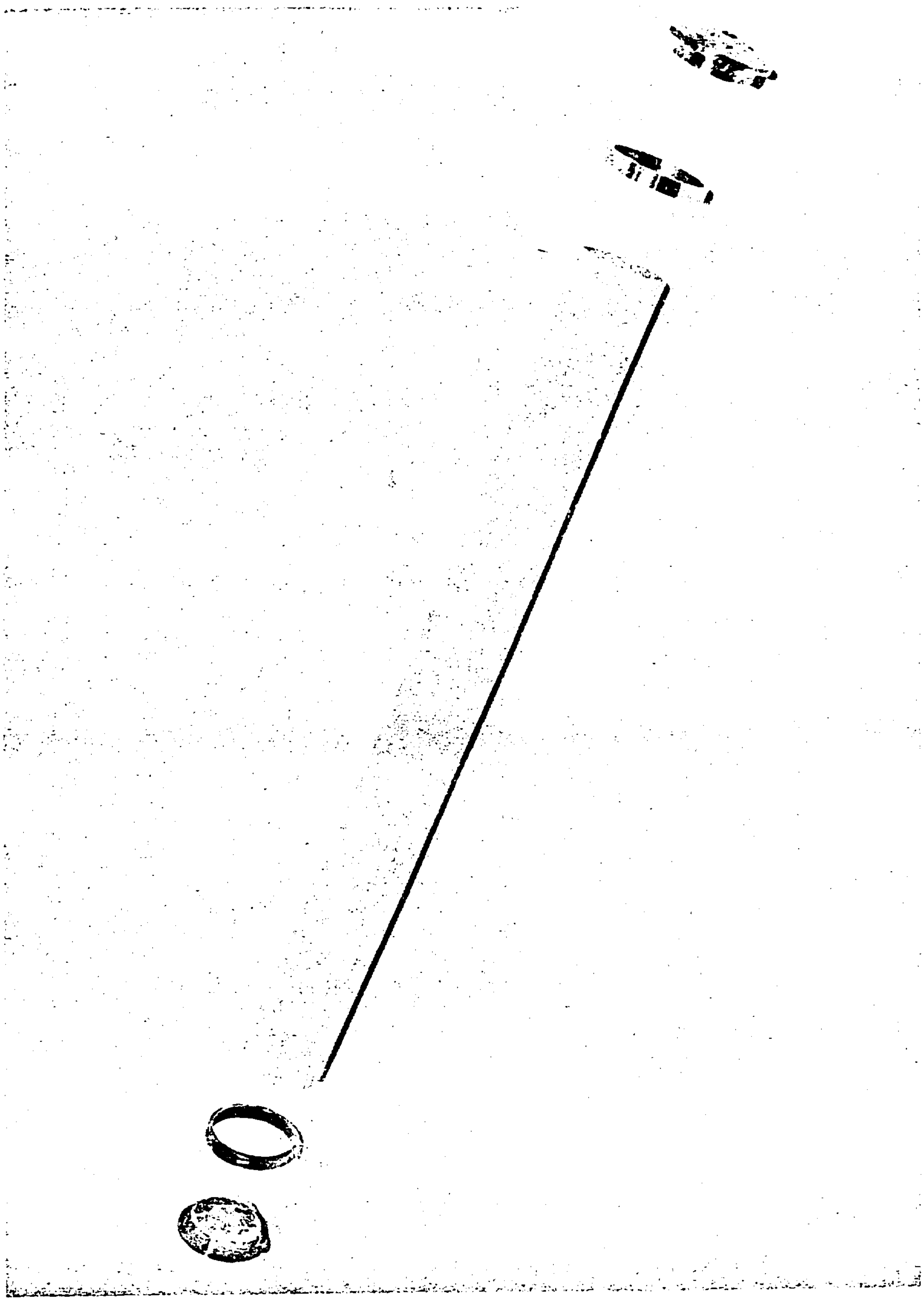

Figure 28 - Buckling Tube Showing End Fittings

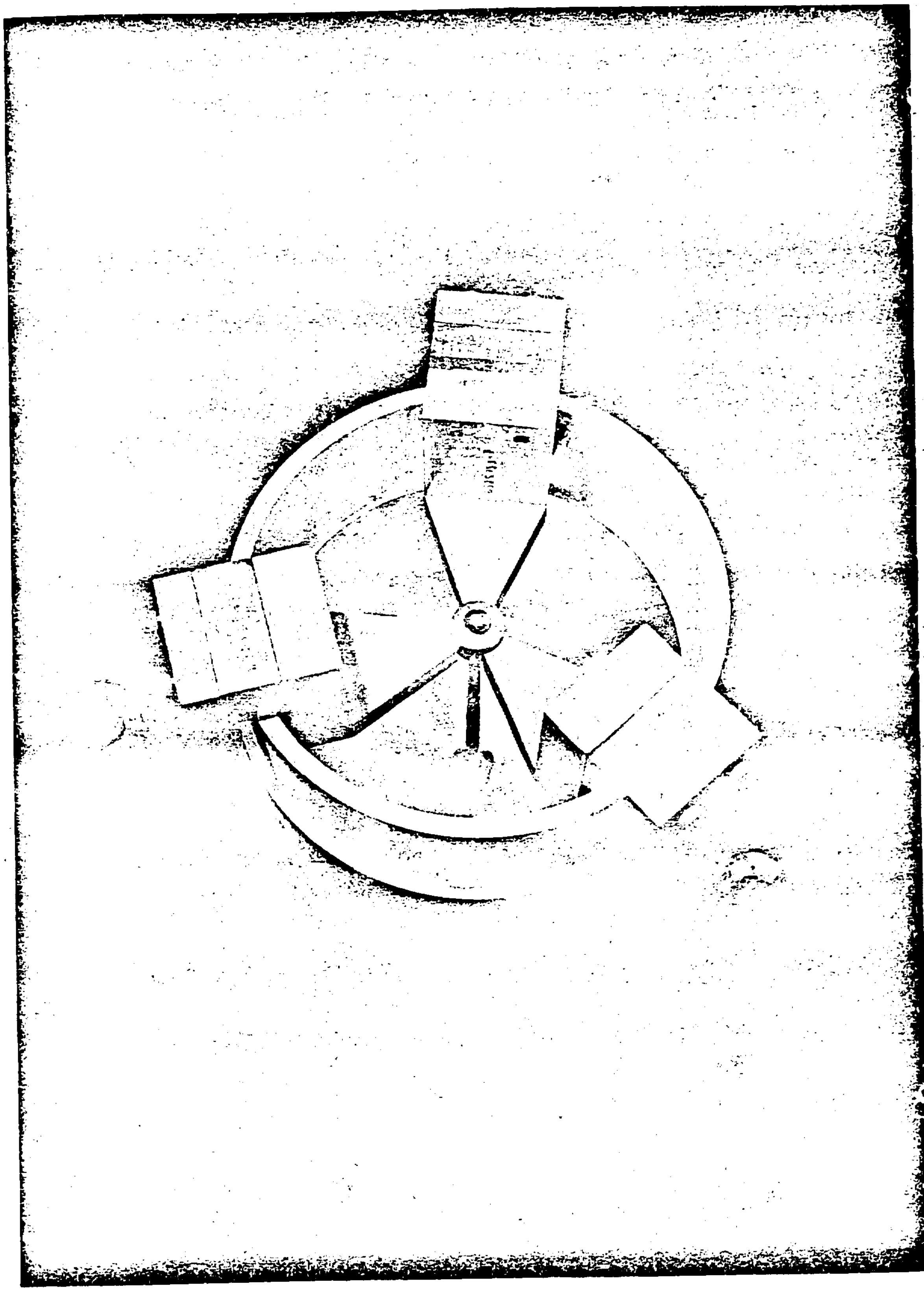

Figure 29 - Buckling Rod Specimen in Cryostat with Centering Ring

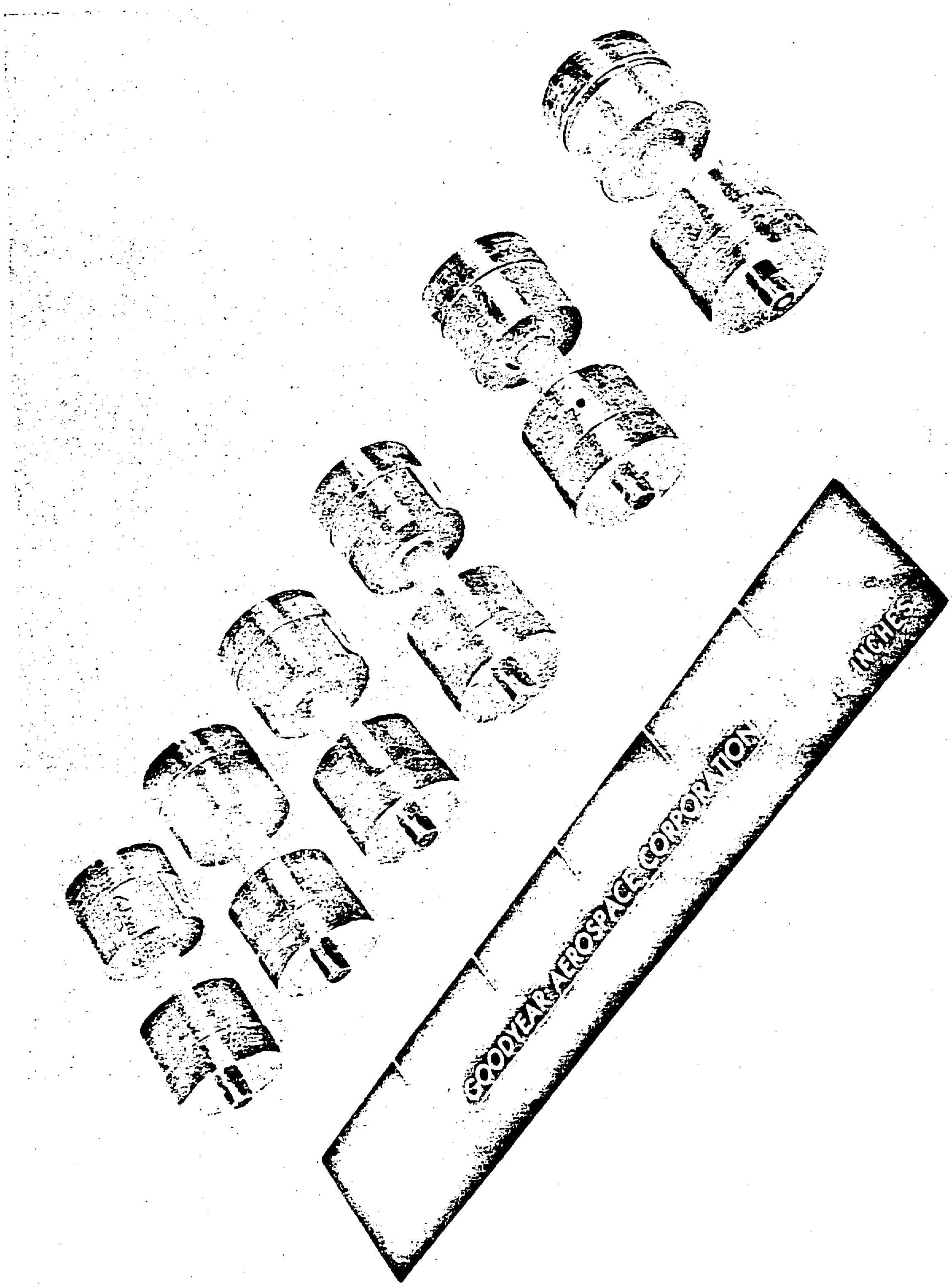

Figure 30 - Compression Rod Specimens before Test

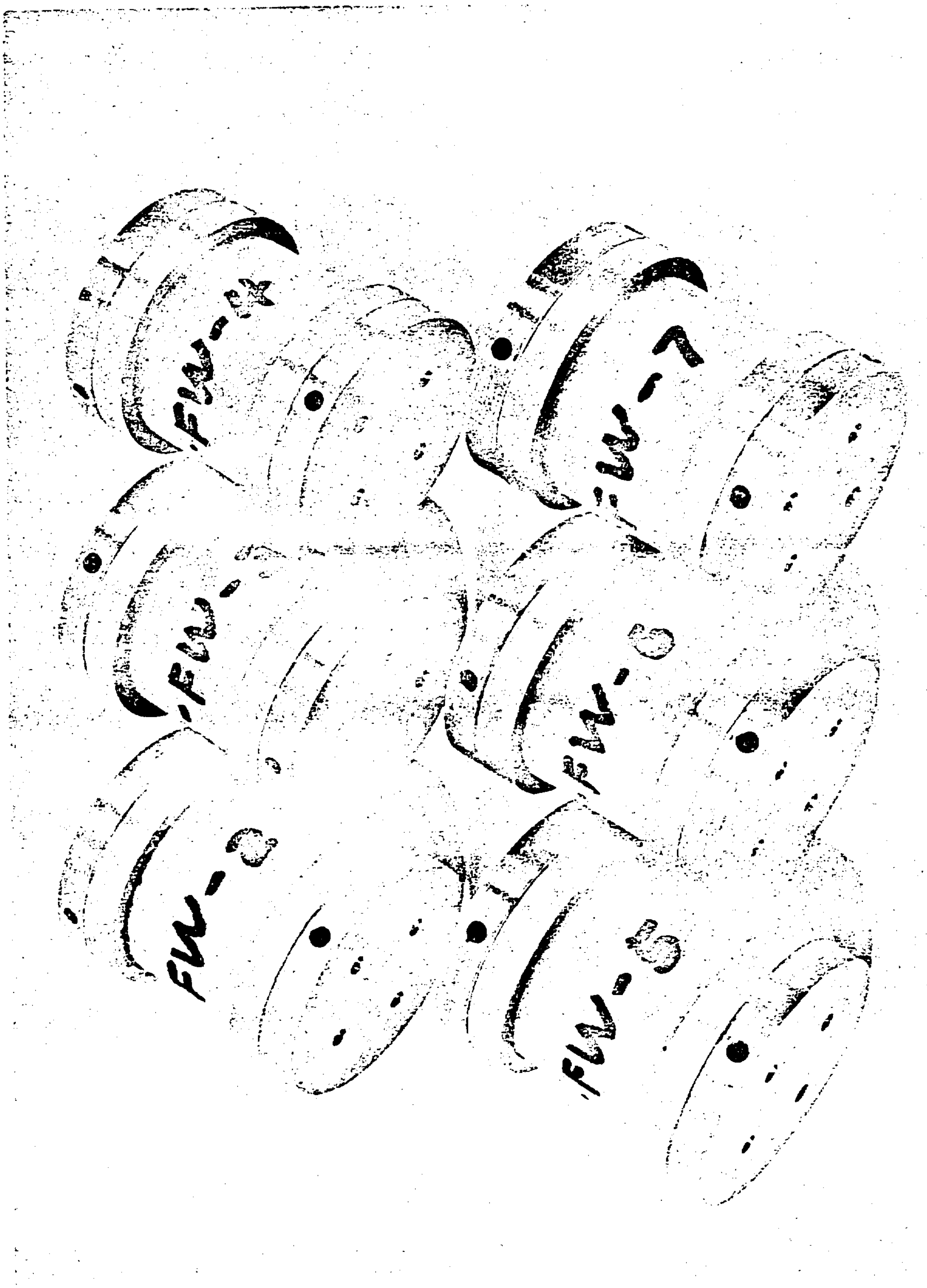

Figure 31 - Compression Tube Specimens before Test

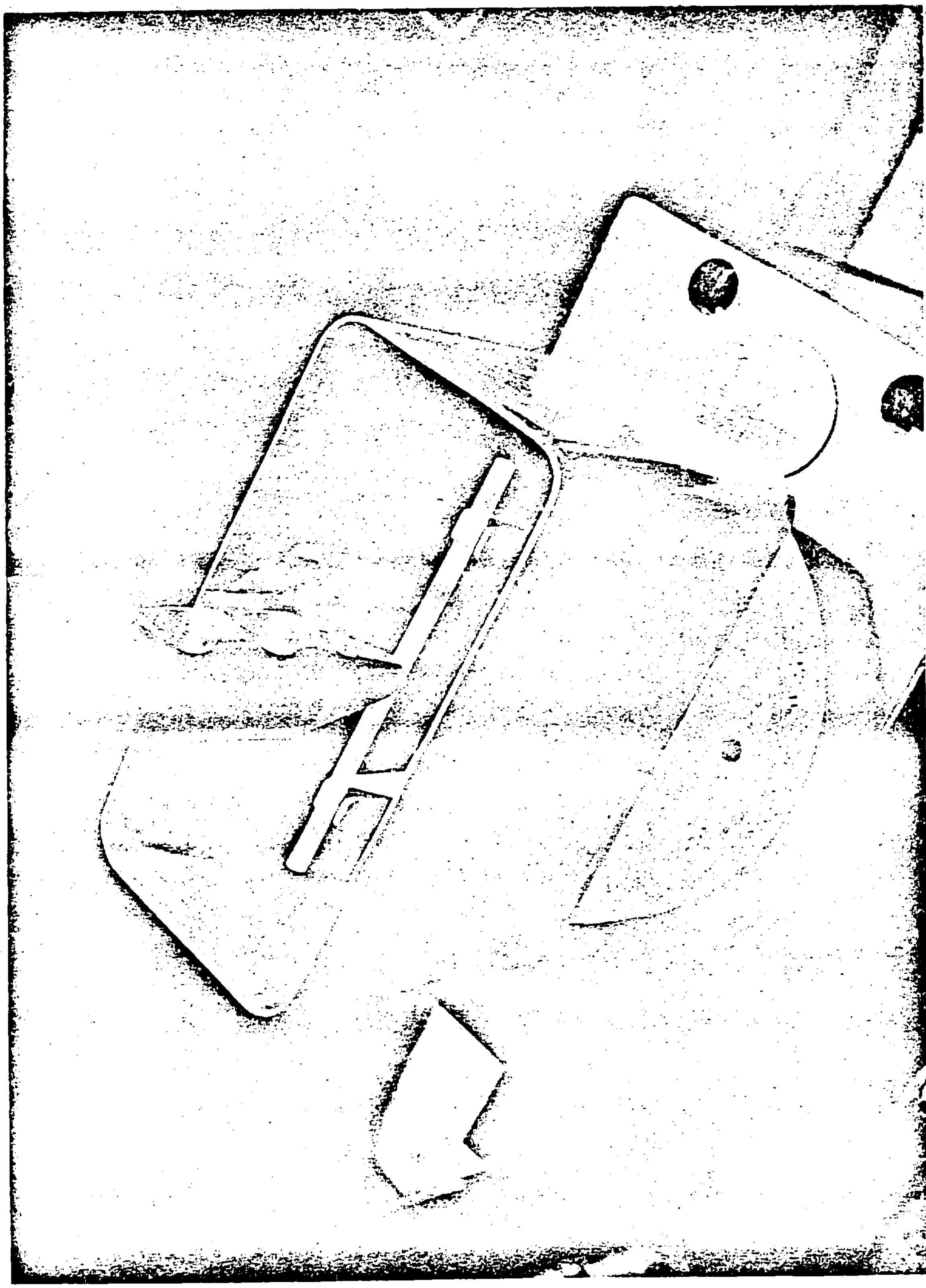

Figure 32 - Flexural Rod in Cryostat before Test

in Figure 33. All of the specimens were tested in either an Instron testing machine or a Baldwin testing machine, depending on the availability and loads required. The rate of loading for each test was selected so that failure of the specimen would occur within a time span of two to three minutes, the same as for the mechanical properties testing program. The test temperature of 77 K was obtained by submerging the assembled test specimen in liquid nitrogen contained in a cryostat during the entire test.

Material Selection

The cryogenic test methods development and the environmental effects assessments were conducted using the following materials:

1. Reinforcement materials
   a. Style 1543 S/901 glass
   b. Style 1581 S/901 glass
   c. Unidirectional 20-end roving S/901 glass
   d. Cross-plied 20-end roving S/901 glass in a 1:1 layer dispersion
2. Resin - U. S. Polymeric E-787 resin system in accordance with Specification WS 1028A. This resin system was used in prepreg form with each of the above reinforcements.

The materials selected represent both filament-wound roving and glass-cloth fabric impregnated with the epoxy resin system that has been proved successful on the Polaris A-3, first-stage, reinforced plastic motor case

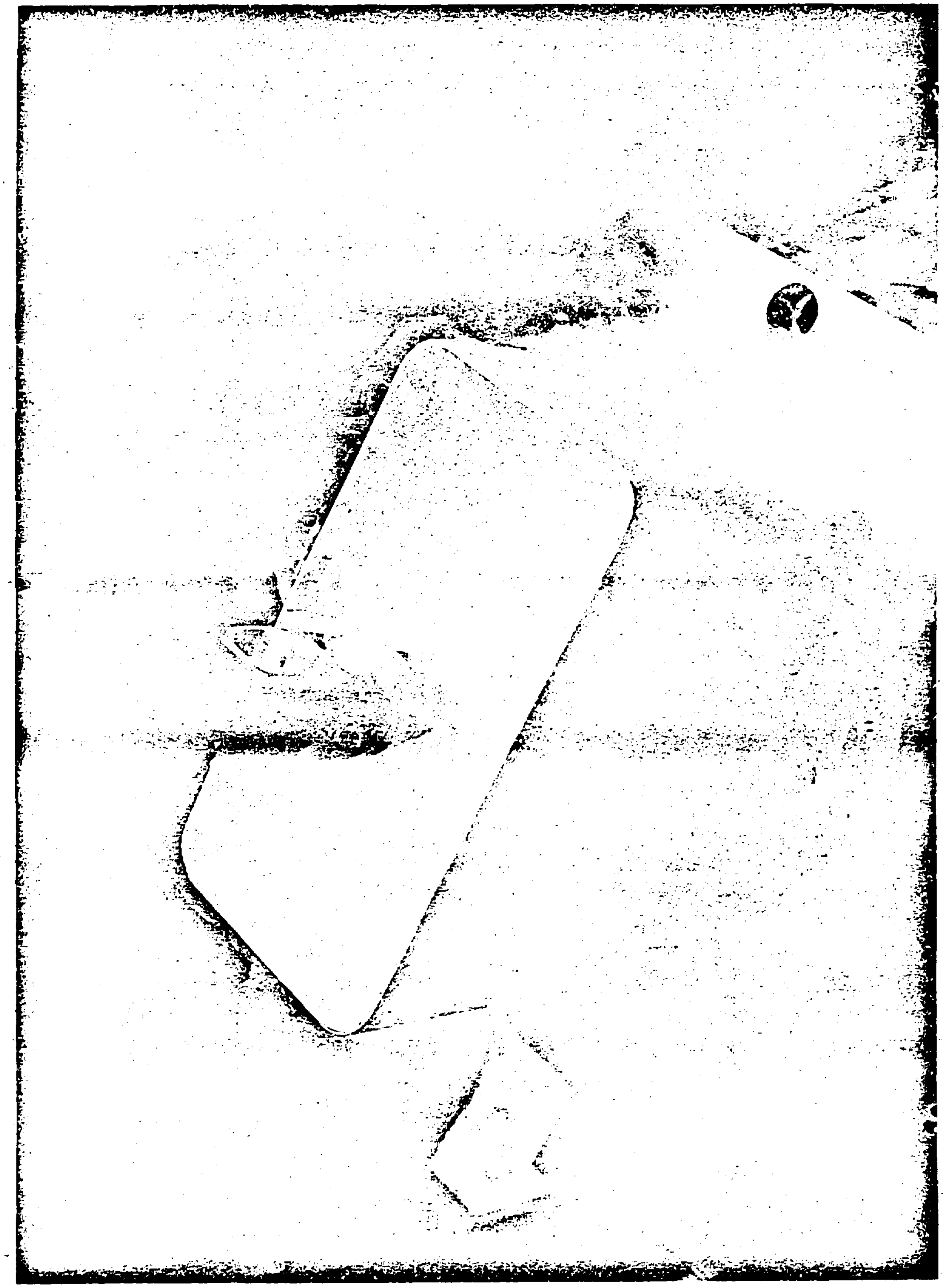

Figure 33 - Flexural Rod in Cryostat with Liquid Nitrogen

program. The E-787 resin system consists of 50 parts by weight (pbw) Epon resin 828, 50 pbw Epon resin 1031, 90 pbw NMA curing agent, and 0.55 pbw BDMA accelerator. The glass used in the materials selected was the higher strength S glass developed by Owens-Corning Fiberglas Corporation. These materials were selected on the basis that they were the most representative of the currently available, reinforced plastic materials that could be used in structural design of actual hardware for use in cryogenics application.

In the final phase of the program, the testing was expanded to include additional reinforcements and resins showing promise of use at cryogenic temperatures. This permitted the application of the previously developed test specimens and test techniques to new families of materials for obtaining a further measure of their validity. The major systems selected were:

1. A chemically pure epoxy resin with an aromatic amine curing agent (100 pbw DER 332/28 pbw DEH 50) and S glass reinforcement
2. A chemically pure epoxy resin system with a Lewis acid curing agent (100 pbw DER 332/3 pbw BF_3-MEA) and S glass reinforcement
3. A new polyester resin system (100 pbw Selectron 5158/1 pbw t-butyl perbenzoate) and S glass reinforcement

The first and third systems were wet systems and the second system a preimpregnated system. The laminates used in each system were cross-plied with 20 end roving in a 1:1 layer dispersion, with the epoxy resins reinforced with an S/901 glass and the polyester resin reinforced with an S glass with a polyester compatible sizing.

A limited study of two other systems was also included in the third year's effort. One system employed Pittsburgh Plate Glass 1062 15-end roving with the E-787 resin system, and the other employed silicon carbide whiskers with the S glass and DER 332/DEH 50 epoxy resin system. The laminates of each system consisted of cross-plied roving in a 1:1 layer dispersion.

Test Results

General

The primary objective of this program was the establishment of systematized procedures to yield reliable data for the prediction of mechanical performance of reinforced plastic materials when employed in components for cryogenic service. The formal test portion of the program was to perform the task of sampling these test techniques and methods on various plastic laminates. Therefore, the test results are not to be viewed as complete data for design purposes, but merely as a start toward this end.

Mechanical Properties Testing

The mechanical properties testing investigations reported in this paper can be divided into three parts. The first part included the testing of four materials in three directions and at four test temperatures. The following properties were evaluated at 298, 197, 77, and 20 K:

1. Tensile ultimate strength, notched strength, elongation, hysteresis, and modulus of elasticity
2. Ultimate compressive strength
3. Flexural strength
4. Interlaminar shear strength (guillotine test method)
5. Bearing strength

The materials tested all contained S/HTS glass reinforcement and U.S. Polymeric E-787 epoxy resin, and included a unidirectional filament-wound (UFW) roving laminate, a bidirectional filament-wound (BFW) roving laminate, a basically unidirectional cloth laminate (1543), and a bidirectional cloth laminate (1581). The second part included the testing of three materials in three directions and at three test temperatures. The properties evaluated were the same as before and tests were conducted at 298, 77, and 20 K. The laminates tested were all bidirectional filament wound panels using S/HTS glass roving with three different resin systems:

1. DER 332/BF_3 epoxy
2. DER 332/DEH 50 epoxy
3. Selectron 5158 polyester

The third part included the testing of three materials at two test temperatures. The properties evaluated for this part at 298 and 77 K were:

1. Tensile ultimate strength, elongation, and modulus of elasticity
2. Ultimate compressive strength
3. Flexural strength
4. Flexural shear strength
5. Bearing strength

The three materials tested were all bidirectional filament-wound laminates using the following materials:

1. S/HTS glass, DER 332/DEH 50 epoxy resin, and 1.5 percent by weight of silicon carbide whiskers

2. S/HTS glass, DER 332/DEH 50 epoxy resin, and 2 percent by weight of silicon carbide whiskers
3. Pittsburgh Plate Glass 1062 glass and E-787 epoxy resin

All specimens in this part were tested by loading in the parallel direction only, whereas in the previous two parts the loading directions included the parallel, 45 deg, and normal directions. The parallel direction is considered the direction parallel to the warp direction for cloth or the direction of the first layer for the filament-wound panels. The other two loading directions are 45 deg and 90 deg (normal) to this direction. The average test results for these three phases are included in Tables 1, 2, and 3.

Structural Model Testing

The ultimate demonstration is the ability to achieve, in structural shapes, the properties previously obtained in the mechanical properties testing program. The tabulated data of the results are given in Table 4. This table includes the average results for each material at each test temperature, and the expected results as determined from the mechanical properties testing program. The values obtained indicate good agreement with the mechanical testing data, and show that the mechanical properties values obtained in the previous testing portion of the program are reproducible in actual structural shapes as long as the fabrication process is comparable.

Analysis of Test Results

General

The results of the reported effort must be analyzed in light of the overall objective, which was the development of a systematized procedure to yield

TABLE 1 - AVERAGE TEST RESULTS - LOAD PARALLEL

TO REINFORCEMENT (Sheet 1 of 2)

Test	Test Temp, °K	1543 S Glass E-787	1581 S Glass E-787	UFW S Glass E-787	BFW S Glass E-787
Flexural Strength, psi	298 197 77 20	133 264 167 701 207 525 217 897	104 186 134 650 170 638 180 325	223 711 309 289 468 554 496 104	170 600 232 401 251 753 216 697
Tensile Strength, psi	298 197 77 20	169 316 186 190 232 385 220 683	92 506 115 540 144 543 137 770	280 890 337 038 330 628 297 741	147 360 173 121 188 403 168 993
Compressive Strength, psi	298 197 77 20	89 503 106 620 122 448 128 171	62 587 79 663 103 202 109 097	151 209 173 089 237 646 235 666	92 348 117 400 127 509 129 161
Bearing Yield, psi	298 197 77 20	44 441 47 675 79 083 79 842	37 980 41 458 64 225 71 625	50 091 60 516 69 125 67 975	42 268 56 141 77 741 68 108
Shear, psi ●	298 197 77 20	8 045 9 923 10 137 10 299	7 912 10 034 11 206 10 074	8 099 7 573 8 030 8 869	5 325 7 260 6 772 6 689
Notched Tensile Strength, psi	298 197 77 20	143 323 176 570 207 076 218 962	71 360 87 944 114 433 118 026	277 256 299 645 307 880 291 478	147 833 190 247 193 139 185 422
Initial Tensile Modulus, psi	298 197 77 20	5 495 000 5 565 830 5 858 330 5 997 857	3 286 670 3 496 330 3 980 000 4 266 000	8 184 000 9 082 100 8 833 000 9 550 800	5 050 000 6 095 000 6 200 000 6 226 000
Ultimate Tensile Elongation, percent	298 197 77 20	3.27 3.51 4.48 4.09	3.37 4.11 5.01 4.81	4.40 4.40 5.30 5.10	3.62 5.33 4.55 4.43
Average Resin Content, percent		32.59	37.69	18.03	19.10
* Average Density, lb/cu in.		.0668	.0639	.0721	.0694

● Guillotine shear test used except where indicated by *, which denotes flexural shear test.

TABLE 1 - AVERAGE TEST RESULTS - LOAD PARALLEL TO REINFORCEMENT (Sheet 2 of 2)

Material					
BFW S Glass DER 332/ BF_3	BFW S Glass DER 332/ DEH 50	BFW S Glass Selectron 5158	BFW S Glass DER 332/ DEH 50 1.5% Whiskers	BFW S Glass DER 332/DEH 50 2% Whiskers	BFW 1062 Glass E-787
176 835 260 225 231 037	129 601 201 091 187 253	144 231 163 599 153 999	110 606 184 038	111 990 170 108	141 415 215 750
160 169 174 459 166 617	138 396 172 296 161 105	114 462 154 383 133 082	68 826 101 575	69 138 97 046	91 816 124 480
84 493 129 011 138 828	80 792 105 939 118 317	73 021 91 468 92 837	83 781 123 128	87 534 139 086	84 080 122 061
45 758 73 370 76 850	40 495 75 000 67 400	34 396 59 241 57 367	34 880 64 400	34 507 64 825	40 155 62 282
3 195 4 286 4 643	5 630 8 179 9 346	3 273 3 407 3 225	6 075* 7 672*	7 477* 7 550*	7 457* 8 691*
158 154 147 544	133 202 154 712	119 523 149 919			
5 795 800 7 029 500 7 000 850	5 212 800 5 527 200 6 710 200	5 863 800 6 091 600 5 961 700	4 505 900 5 275 250	4 214 550 5 122 250	5 061 600 5 799 750
3.54 3.84 4.18	3.32 3.90 3.94	3.05 3.98 3.58	1.89 2.48	1.99 2.84	2.66 4.22
17.00	18.88	18.15	26.51	30.25	17.31
.0748	.0712	.0755	.0662	.0657	.0765

TABLE 2 - AVERAGE TEST RESULTS - LOAD 45 DEG TO REINFORCEMENT

Test	Test Temp, °K	Test Material: 1543 S Glass E-787	1581 S Glass E-787	UFW S Glass E-787	BFW S Glass E-787	BFW S Glass DER 332/BF_3	BFW S Glass DER 332/DEH 50	BFW S Glass Selectron 5158
Flexural Strength, psi	298	44 824	48 529	22 004	41 242	38 798	35 894	28 619
	197	61 222	72 274	25 168	58 151	--	--	--
	77	75 756	105 156	25 298	72 722	58 164	57 380	52 736
	20	81 668	103 330	31 591	66 406	--	--	--
Tensile Strength, psi	298	19 937	32 716	--	21 211	16 973	19 114	16 803
	197	23 607	46 034	--	23 298	--	--	--
	77	27 861	50 684	--	30 354	25 637	27 739	18 352
	20	27 490	46 613	--	31 625	--	--	--
Compressive Strength, psi	298	31 245	31 370	23 180	25 899	26 705	24 907	19 052
	197	45 471	45 939	40 650	39 050	--	--	--
	77	66 456	69 935	63 705	57 365	56 694	51 798	42 453
	20	68 476	77 689	63 500	60 093	--	--	--
Notched Tensile Strength, psi	298	25 533	37 200	--	29 252	--	--	--
	197	30 396	53 080	--	30 834	--	--	--
	77	36 783	59 689	--	40 949	--	--	--
	20	35 779	56 741	--	41 027	--	--	--
Initial Tensile Modulus, psi	298	1 894 670	1 498 670	--	3 060 000	3 255 900	1 864 400	3 615 200
	197	2 154 000	2 108 670	--	4 060 000	--	--	--
	77	3 011 330	2 730 670	--	4 700 000	5 119 300	4 058 400	4 640 000
	20	3 531 330	2 834 000	--	4 761 600	--	--	--
Ultimate Tensile Elongation, Percent	298	3.27	>10.0	--	10.10	2.67	5.69	10.00
	197	4.47	10.57	--	2.00	--	--	--
	77	1.11	6.71	--	1.09	0.67	1.02	0.85
	20	1.21	4.84	--	1.02	--	--	--
Average Resin Content, Percent		32.59	37.69	18.03	19.10	17.00	18.88	18.15
Average Density, lb/cu in.		.0668	.0639	.0721	.0694	.0748	.0712	.0755

TABLE 3 - AVERAGE TEST RESULTS - LOAD NORMAL TO REINFORCEMENT

Test	Test Temp, °K	Test Material: 1543 S Glass E-787	1581 S Glass E-787	UFW S Glass E-787	BFW S Glass E-787	BFW S Glass DER 332/BF_3	BFW S Glass DER 332/DEH 50	BFW S Glass Selectron 5158
Flexural Strength, psi	298	43 665	96 297	12 336	150 447	129 963	114 732	117 080
	197	55 480	126 103	17 773	171 359	--	--	--
	77	76 035	157 132	11 951	167 698	165 942	147 682	117 925
	20	74 688	170 221	13 378	157 461	--	--	--
Tensile Strength, psi	298	28 613	84 474	5 995	--	145 353	124 360	125 103
	197	35 008	98 752	--	--	--	--	--
	77	42 190	128 010	--	--	158 282	163 116	159 379
	20	37 554	118 110	--	--	--	--	--
Compressive Strength, psi	298	35 753	59 161	25 291	79 225	80 404	77 700	62 865
	197	55 480	74 089	37 165	109 176	--	--	--
	77	69 359	94 826	51 768	99 330	112 369	88 640	71 854
	20	65 480	100 847	52 176	102 414	--	--	--
Notched Tensile Strength, psi	298	24 033	67 932	--	--	137 872	127 160	118 340
	197	29 595	80 555	--	--	--	--	--
	77	36 497	100 281	--	--	133 159	141 568	153 818
	20	36 218	104 686	--	--	--	--	--
Initial Tensile Modulus, psi	298	2 144 670	3 166 000	--	--	5 112 200	4 699 600	5 056 000
	197	2 463 330	3 340 670	--	--	--	--	--
	77	3 237 330	3 974 000	--	--	5 730 300	5 368 900	4 893 500
	20	3 287 330	4 178 000	--	--	--	--	--
Ultimate Tensile Elongation, Percent	298	3.71	3.30	--	--	3.85	3.22	3.02
	197	4.98	3.96	--	--	--	--	--
	77	5.58	4.64	--	--	4.13	4.22	4.48
	20	5.68	4.50	--	--	--	--	--
Average Resin Content, Percent		32.59	37.69	18.03	19.10	17.00	18.88	18.15
Average Density, lb/cu in.		.0668	.0639	.0721	.0694	.0748	.0712	.0755

TABLE 4 - AVERAGE RESULTS OF MODEL TESTING

FOR S GLASS AND E-787 RESIN

Test	Configuration	Laminate Form	Test Temp, °K	Average Result, psi	Expected Properties, psi
Compression	Tube	BFW	298	68 933	79 225
			77	99 050	99 330
		1543	298	47 367	89 503
			77	78 250	122 448
		1581	298	34 820	62 587
			77	55 600	103 202
	Rod	UFW	298	147 719	151 209
			77	277 185	237 646
Tension	Tube	BFW	298	111 067	127 200
			77	101 300	162 800
		1543	298	143 233	169 316
			77	183 300	232 385
		1581	298	66 633	92 506
			77	103 266	144 543
	Rod	UFW	298	309 229	294 928
			77	323 486	330 628
Flexure	Rod	UFW	298	253 933	223 711
			77	417 250	468 554
Buckling	Tube	BFW	298	45 083	37 600
			77	62 167	41 550
		1543	298	41 653	45 750
			77	45 750	48 650
		1581	298	29 140	28 500
			77	33 140	32 360
	Rod	UFW	298	21 090	9 900
			77	31 137	9 870

reliable test data for reinforced plastic materials at cryogenic temperatures. Such a procedure must meet the following requirements:

1. A complete knowledge of the raw ingredients that make up any test panel
2. A fabrication process that is representative of methods that would be employed in actual parts
3. Test specimens that are both simple and economical to fabricate, yet are easily reproduced
4. The test procedures and fixtures must be both simple and economical
5. The mode of failure of the mechanical properties test specimens must be indicative of the desired results
6. The scatter of the test results should be minimum
7. The test data must be applicable to actual structures

One of the major challenges was the achievement of the correct mode of failure for all specimens in mechanical testing. The use of the filament-wound roving laminates increased the difficulties of this requirement because of its high tensile and compressive strength, yet low shear strength. The flexure and bearing specimens were standard specimens, and there were no problems in achieving the correct mode of failure for these specimens. The tension, compression, and shear specimens posed more of a problem, however, as can be seen in Figures 34, 35, and 36. The correct mode of failure was also achieved in these specimens.

Test Evaluation

The mechanical properties testing program, although not to be viewed as

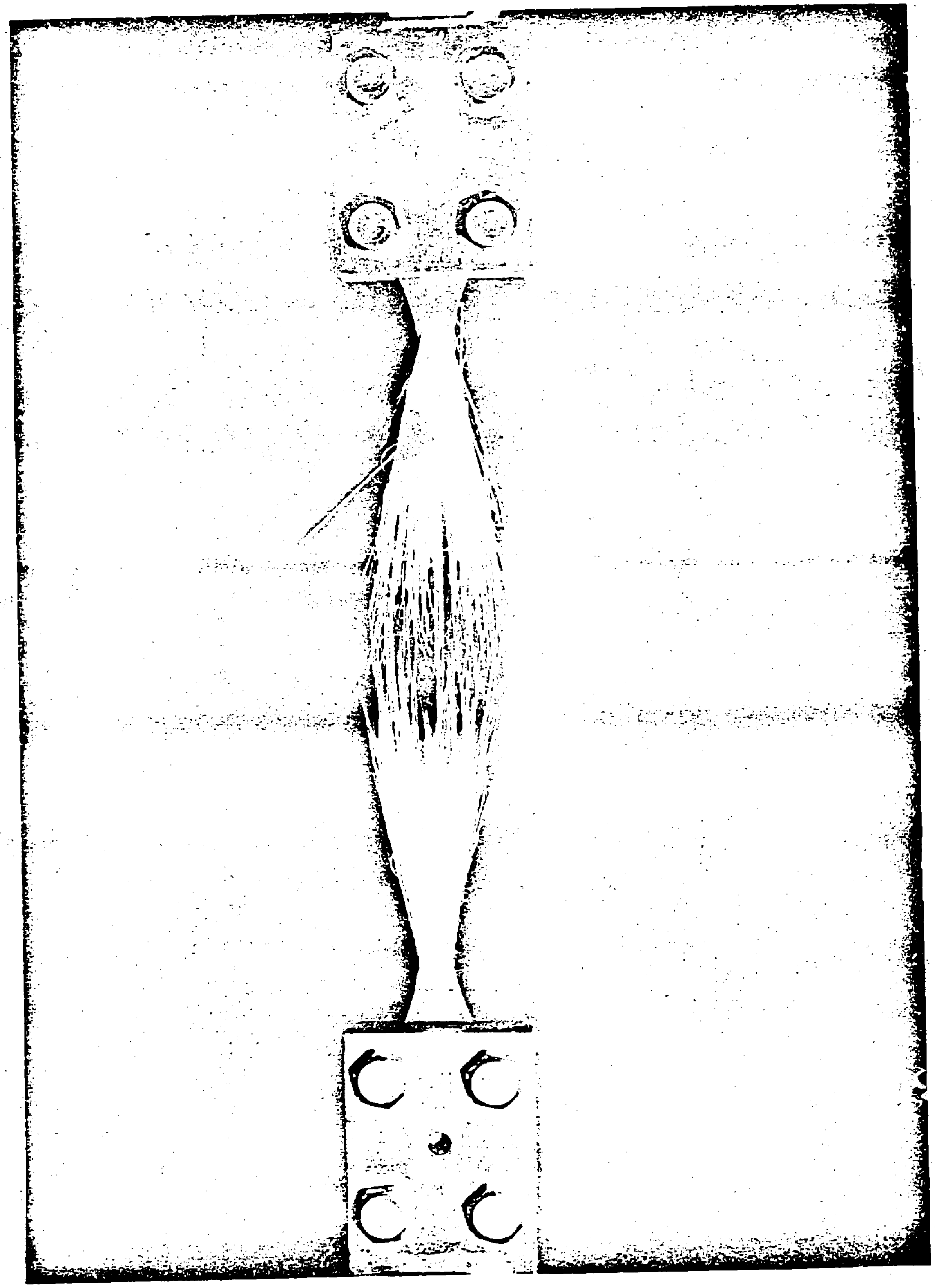

Figure 34 - Typical Failure of Unidirectional Filament-Wound Tensile Specimen

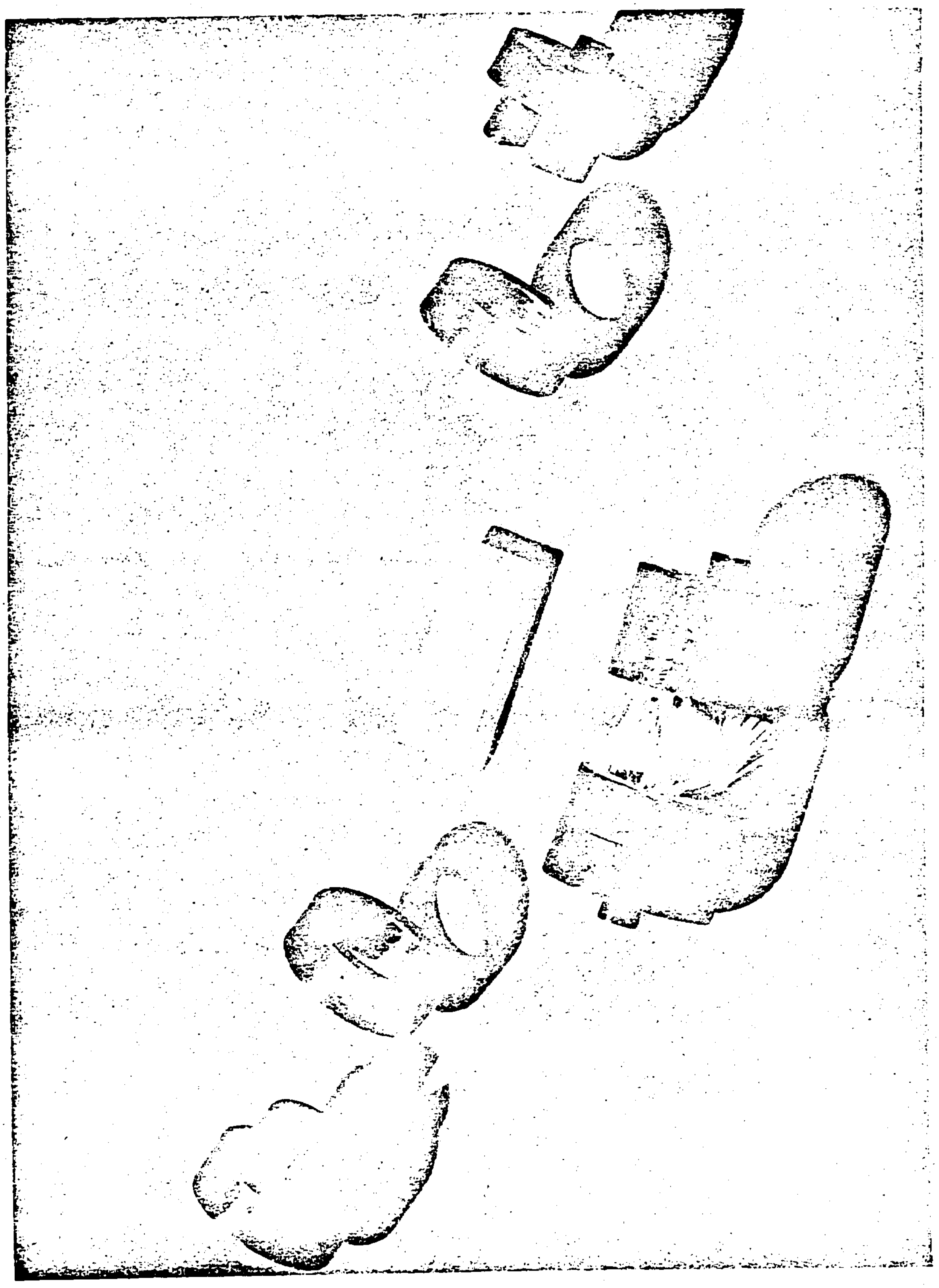

Figure 35 - Typical Failure of Compression Specimen, Load Block, and Retaining Ring before Assembly

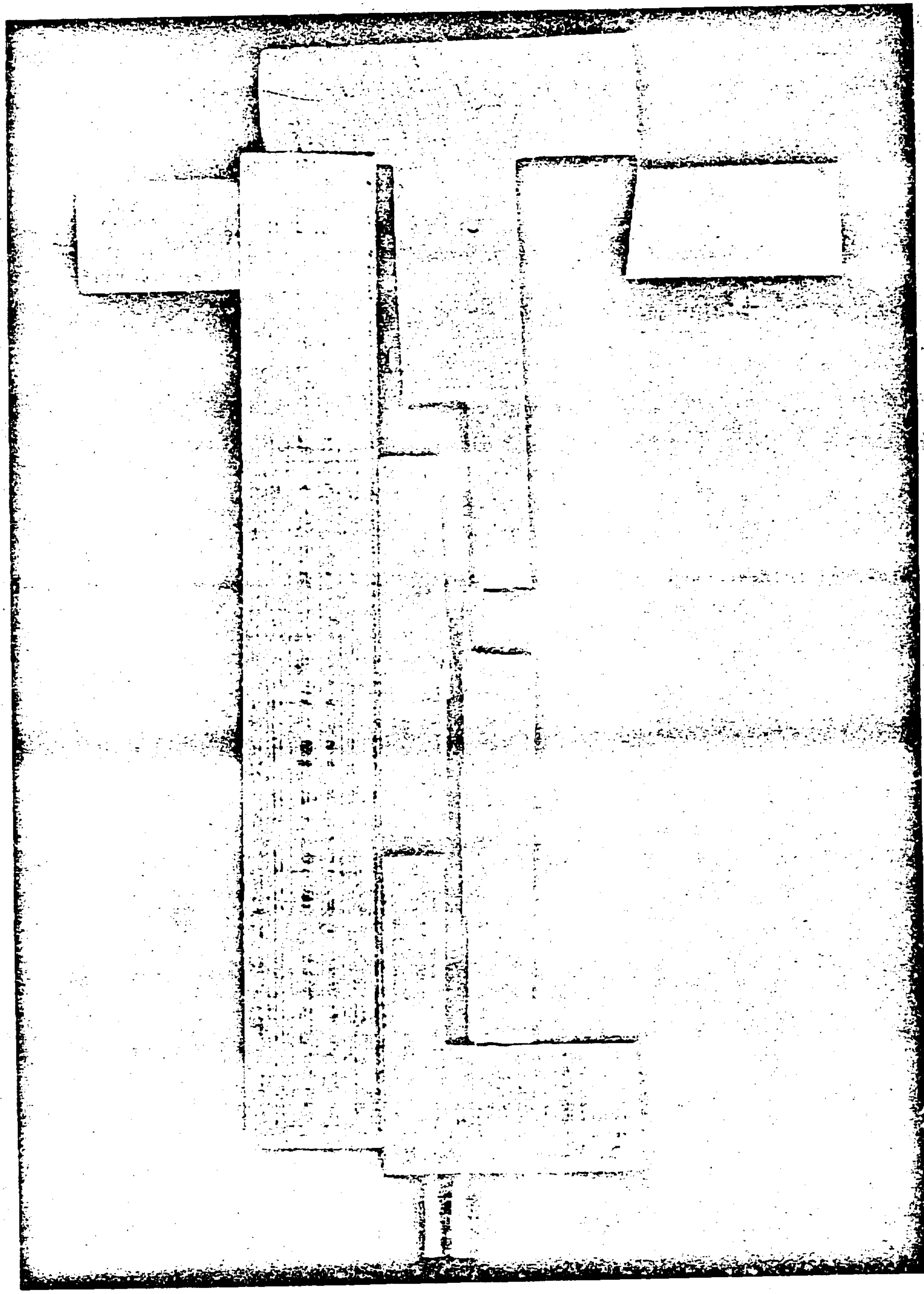

Figure 36 - Typical Failed Specimen after Guillotine Shear Test

providing complete data for design purposes, has indicated that fiberglass-reinforced plastics should become a material of increased use in structural application at cryogenic temperatures. Not only did the fiberglass specimens maintain their integrity at these lower temperatures, but the mechanical properties increased substantially over the corresponding room temperature properties. The fact that the results of the mechanical properties testing program were reproducible in the structural models, and that the artificial weathering tests showed no detrimental effects, further substantiates the potential of these materials.

An evaluation of the various systems tested would indicate that the S glass and U.S. Polymeric E-787 epoxy resin system develops the most optimum overall properties. The S glass and DER 332/BF_3 epoxy resin system gave comparable results with the exception of interlaminar shear strength, which was low. The 1062 glass and E-787 epoxy resin system is competitive from a strength-cost standpoint.

The addition of approximately 6 percent silicon carbide whiskers to the DER 332/DEH 50 epoxy resin (2 percent of the total laminate) resulted in a seven-percent increase in compressive strength and a 20-percent increase in shear strength. However, because of the abrasive effect of the whiskers on the glass during winding, the tensile strength was greatly reduced. These initial efforts with whiskers have been encouraging.

Conclusions

Significant progress has been made toward the ultimate goal of obtaining a handbook of design data for reinforced plastics at cryogenic temperatures. New testing techniques, procedures, and specimen configurations were developed where necessary for the purpose of upgrading and improving the validity and direct design utility of the test data. Selected materials have been tested and all pertinent mechanical and thermophysical properties of all of the specified cryogenic temperatures have been achieved. The model testing has shown that these test values apply not only to these test specimens, but also to actual structural parts fabricated of these materials.

Conclusions reached as a result of the testing program may be summarized as follows:

1. The modes of failure of the test specimens in all tests and the results of the model testing show that valid data have been obtained.

2. The scatter in the test results has been reasonably small, considering the normal scatter in the raw materials properties.

3. It has been demonstrated that testing of plastic specimens, sufficiently large in size to yield usable design data at cryogenic temperatures, can be accomplished at reasonable cost if proper specimen configuration, test techniques, and test procedures are employed.

4. Further verification of the potential use of fiberglass-reinforced plastics for structural applications at cryogenic temperatures has been achieved. Not only did the fiberglass laminates maintain their integrity, but also their mechanical properties increased in value at cryogenic temperatures. Fiberglass structures, with their high strength-to-weight ratios, will see increased use.

5. The relatively low thermal contraction and conductivity of fiberglass laminates would make the use of these materials desirable as structural members where the consideration of heat sink and thermal stresses are of major importance.